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# ARGONNE NATIONAL LABORATORY

Advanced Photon Source | Center for Nanoscale Materials

# 2015 APS/CNM USERS MEETING

## PROGRAM AND ABSTRACTS

# 2015 APS/CNM Users Meeting

# PROGRAM AND ABSTRACTS

# User Facilities at Argonne National Laboratory

## User Contacts

### Advanced Photon Source

http://www.aps.anl.gov
630-252-9090
apsuser@aps.anl.gov

### Argonne Leadership Computing Facility

http://www.alcf.anl.gov
630-252-0929

### Argonne Tandem Linac Accelerator System

http://www.phy.anl.gov/atlas
630-252-4044

### Center for Nanoscale Materials

http://nano.anl.gov
630-252-6952
cnm_useroffice@anl.gov

# Table of Contents

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CNM: Steve Smith and Seungbum Hong

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Matthew Grayson, Electrical Engineering & Computer Science, Northwestern University

Axel Hoffmann, Materials Science Division, Argonne National Laboratory

Seungbum Hong, Nanoscience & Technology Division, Argonne National Laboratory (Chair)

Yasuo Ito, Dept. of Physics, Northern Illinois University

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Steve Smith, Dept. of Physics, South Dakota School of Mines & Technology (Vice Chair)

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## 2015 Users Meeting Organizing Committee

Deena Blair— General support

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Jim Corsolini (CEP) — Video support

Jay Fisher (CEP) — Video support

Beverly Knott — General support

Jacquelin LeBreck (CEP) —General support

Linda Conlin/Michele Nelson (CEP) — Design and production of program book

Rachel Reed — Signage, poster session, social events support, APS Users Organization arrangements, meeting coordination

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Tara Videtic — Site access

Carmie White, Argonne Guest House — On-site logistics and amenities coordination

Karin Widuch — Reimbursements, travel arrangements, budget tracking, poster session

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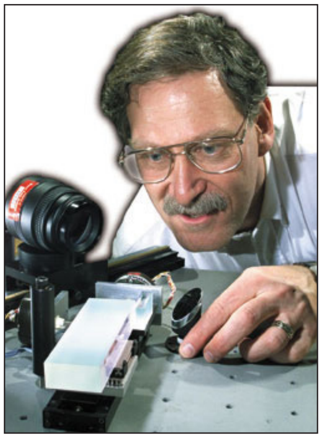
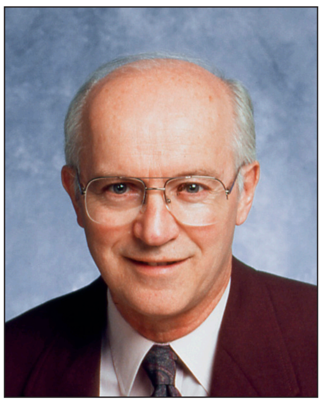
# 2015 Arthur H. Compton Award

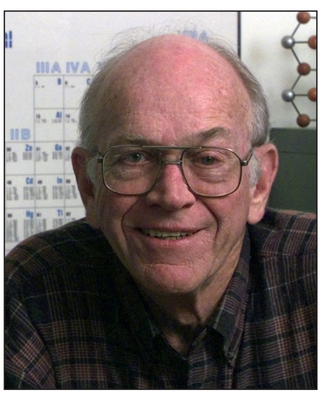
The 2015 APS Arthur H. Compton Award has been awarded to Gene E. Ice, Bennett C. (Ben) Larson, and Cullie J. Sparks (posthumously), all of Oak Ridge National Laboratory, for seminal developments that have advanced capabilities for spatially and temporally resolved synchrotron x-ray research.

The nomination recognizes transformative breakthroughs in three areas: (1) techniques for using single synchrotron pulses for ultrafast time-resolved diffraction studies; (2) focusing monochromators for producing small, intense synchrotron x-ray beams; and (3) x-ray microscopy with three-dimensional, submicron spatial resolution. The impact of these breakthroughs has been important not only at the Advanced Photon Source but also at light sources worldwide.

The three researchers exploited the high brilliance and intrinsic pulsed time-structure of synchrotron sources to create techniques and instrumentation that are now considered indispensible tools for x-ray science. "With their vision, their deep understanding of the interaction of x-rays with matter, and their exquisite experimental skills, they paved the way to the modern instrumentation of today's large-scale x-ray facilities," said Helmut Dosch, chair of the board of directors of DESY in Hamburg, Germany.







Top to bottom: Gene E. Ice, Bennett C. Larson, and Cullie J. Sparks

## Time-resolved studies

Ben Larson and his colleagues were the first to couple single synchrotron x-ray pulses with pulsed-laser excitations to perform single x-ray pulse x-ray diffraction measurements. This strategy yielded nanosecond time resolution for materials studies. Larson's seminal experiments [1,2] clarified the mechanism of pulse-laser-induced melting in silicon, thereby resolving a significant controversy. The technique has been adapted to both polychromatic and monochromatic applications at the APS and enables diffraction and spectroscopy to investigate the evolving structure of materials on ultrashort time scales.

Simple in hindsight, this technique immediately transformed synchrotron time-resolved diffraction from measurements limited by detector gating capabilities and time-average intensities to measurements with a temporal resolution determined by the ~100 picosecond synchrotron pulse widths. In addition, the technique had the inherent advantage of the ultrahigh instantaneous intensities available during individual pulses. This now-common technique represents a core capability at the APS and on third- and fourth-generation pulsed x-ray sources in general, and it has opened the way to studies at femtosecond time scales at free-electron laser facilities.

## Precision focusing optics

Cullie Sparks and Gene Ice pioneered the use of dynamically bent curved crystals to simultaneously focus and monochromatize synchrotron x-rays, a technique called sagittal focusing [3,4]. The result was a small, ultra-intense, and tunable monochromatic beam that made it possible to study a new range of weakly scattering samples.

At the time, the idea that monochromator crystals could be bent with sufficient accuracy to focus beams was considered impractical; the crystals would take a saddle shape (anticlastic bending) instead of a single curve. Sparks and Ice found that they could prevent this distortion by cutting ribs on the back of the crystal parallel to the plane of scattering. Solving the otherwise fatal obstacle of anticlastic bending turned sagittal focusing from a theoretical curiosity into a deployable technology—which has now become standard instrumentation. Optics of this type are in use in at least five bending magnets and four insertion device beamlines at APS and on other beamlines at synchrotrons worldwide, and the impact of focused beams continues to grow for studies in materials structure and dynamics, geophysics, environmental science, biophysics, and protein crystallography [5].

## Three-dimensional x-ray microscopy (3DXM)

Gene Ice and Ben Larson, together with colleagues, made advances on three fronts—optics development, experimental geometry, and pattern analysis techniques—to allow submicron-resolution, three-dimensional mapping of heterogeneous microstructures in single-crystal and polycrystalline materials [6, 7]. The technique makes it possible to nondestructively measure the local structure, crystallographic orientation, grain size, grain morphology, and strain in three dimensions with a resolution of less than one micron (micrometer) over areas ranging from a fraction of a micron to hundreds of microns.

Known also as differential aperture x-ray microscopy (DAXM) because of the depth profiling principle, 3DXM depends on the use of an x-ray–absorbing wire that is moved stepwise across the diffraction pattern. Differences in pixel intensities at each step are analyzed by powerful computer-cluster-based analysis programs. The result is that a full diffraction pattern is extracted from individual submicron-sized volume elements—without rotating the sample. The method was demonstrated in 1999 on sector 7, prototyped at a dedicated facility on sector 34 in 2001, and further developed by the APS into a 3D microprobe facility that is now available to general users.

The possible applications of DAXM are enormous [8], because virtually all technologically important materials and advanced processing techniques are based on the generation of heterogeneous microstructures or heterostructure at interfaces between materials. Thus, applications range from analyzing strains and identifying defects in microelectronic devices, to detailing the processes that occur during forge processing of structural alloys, to drug design, to environmental and geological studies. In the context of the APS, the 3DXM facility at sector 34 provides an important bridge between the nanoscale capabilities at sector 26 and the penetrating, high-energy diffraction three-dimensional microscopy at sector 1. As a result, the APS is able to offer nondestructive 3D microscopy from the nanoscale to the macroscale.

## References


[1] B.C. Larson, C.W. White, T.S. Noggle, and D. Mills, "Synchrotron X-ray Diffraction Study of Silicon during Pulsed-Laser Annealing," *Phys. Rev. Lett.* **48**, 337 (1982).

[2] B.C. Larson, J.Z. Tischler, and D.M. Mills, "Nanosecond Resolution Time-resolved X-ray Study of Silicon during Pulsed Laser Irradiation," *J. Mater. Res.* **1**, 144 (1986).

[3] C.J. Sparks, G.E. Ice, J. Wong, and B.W. Batterman, "Sagittal Focusing of Synchrotron X-ray Radiation with Curved Crystals," *Nucl. Inst. & Meth.* **195**, 73 (1982).

[4] G.E. Ice and C.J. Sparks, "Conical Geometry for Sagittal Focusing as Applied to X-rays from Synchrotrons," *J. Opt. Soc. Am.* **11**, 1265 (1994).

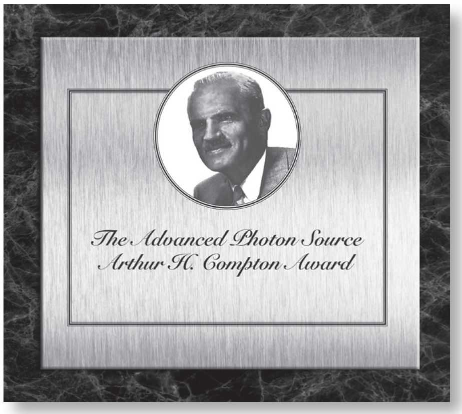
[5] G.E. Ice, J.D. Budai, and J.W.L. Pang, "The Race to X-ray Microbeam and Nanobeam Science" [invited review], *Science* **334**, 1234 (2011).

[6] B.C. Larson, W. Yang, G.E. Ice, J.D. Budai, and J.Z. Tischler, "Three-dimensional X-ray Structural Microscopy with Submicrometre Resolution," *Nature* **415**, 887 (2002).

[7] G.S. Cargill, "Extra Dimension with X-rays," *Nature* **415**, 844 (2002).

[8] B.C. Larson and L.E. Levine, "Submicrometre-resolution Polychromatic Three-dimensional X-ray Microscopy" [invited review], *J. Appl. Crystall.* **46**, 153 (2013).

## About the Award




The Arthur H. Compton award was established in 1995 by the APS Users Organization (APSUO) to recognize an important scientific or technical accomplishment at the Advanced Photon Source. The awards are generally made at APS User Meetings.

Compton was an American physicist who won the Nobel Prize for Physics in 1927 for discovering and explaining changes in x-ray wavelengths resulting from x-ray collisions with electrons, the so-called Compton effect. This important discovery in 1922 confirmed the dual nature (wave and particle) of electromagnetic radiation. A Ph.D. from Princeton University, Compton held many prominent positions including professor of physics at The University of Chicago and chairman of the committee of the National Academy of Sciences that studied the military potential of atomic energy. His position on that committee made Compton instrumental in initiating the Manhattan Project, which created the first atomic bomb.

**Previous award recipients**
Nikolai Vinokurov and Klaus Halbach (1995)
Philip M. Platzman and Peter Eisenberger (1997)
Donald H. Bilderback, Andreas K. Freund, Gordon S. Knapp, and Dennis M. Mills (1998)
Sunil K. Sinha (2000)
Wayne A. Hendrickson (2001)
Martin Blume, L. Doon Gibbs, Denis McWhan, and Kazumichi Namikawa (2003)
Günter Schmahl and Janos Kirz (2005)
Andrzej Joachimiak and Gerold Rosenbaum (2007)
Gerhard Grübel, Simon Mochrie, and Mark Sutton (2009)
Edward Stern, Farrel Lytle, Dale Sayers (posthumously), and John Rehr (2011)
David E. Moncton, John N. Galayda, Michael Borland, and Louis Emery (2013)

# 2015
# APS/CNM Users Meeting

# COMPREHENSIVE PROGRAM

# Monday, May 11

| | |
|---|---|
| 8:00 – 5:00 | Exhibits<br>*Bldg. 402, Gallery (lower level), outside E1100/1200 and Bldg. 402, Atrium* |
| 7:30 – 5:00 | Registration<br>*Bldg. 402, Atrium* |
| 12:00 – 1:30 | Lunch<br>*Tents outside of lower level Gallery* |

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## Opening Session—Morning
## Bldg. 402, Lecture Hall

**Session Chair:** **Eric Landahl (DePaul University) APSUO Steering Committee Chair**

| | |
|---|---|
| 8:50 – 9:00 | Eric Landahl, APSUO Chair<br>*Welcome* |
| 9:00 – 9:20 | Peter Littlewood, ANL Director<br>*Welcome from the Laboratory* |
| 9:20 – 9:40 | Harriet Kung, DOE Office of Basic Energy Sciences<br>*The DOE Perspective* |
| 9:40 – 9:45 | Seungbum Hong, CNM UEC Chair<br>*Introduction of Keynote Speaker* |
| 9:45 – 10:25 | Keynote Speaker: John Rogers, University of Illinois Urbana-Champaign<br>*A Thousand Points of Light – Microscale Light-emitting Diodes for Applications Ranging from Information Display to Optogenetics* |
| 10:25 – 11:00 | Coffee Break (Gallery, outside E1100/1200 and Atrium) |
| 11:00 – 11:25 | Stephen Streiffer, Director, APS<br>*Update on the Advanced Photon Source* |
| 11:25 – 11:45 | Andreas Roelofs, Interim Director, CNM<br>*Update on the Center for Nanoscale Materials* |
| 11:45 – 12:00 | Susan Strasser, APS User Office<br>*Update on NUFO/ARIA* |
| 12:00 | Lunch |

## Parallel Facility Plenary Sessions—Afternoon

## APS Session
## Bldg. 402, Lecture Hall

**Session Chair:** **Don Brown (Los Alamos National Laboratory), APS Users Organization Steering Committee Vice Chair**

| | |
|---|---|
| 1:15 – 1:55 | Keynote Speaker<br>Wenlu Zhu (University of Maryland)<br>*Evolving Rock Structure* |
| 1:55 – 2:35 | Presentation of the 2015 Arthur H. Compton Award<br>*Gene Ice, Bennett Larson, and Cullie Sparks (posthumously)* |
| 2:35 – 2:55 | Invited Student Talk: Andrew Ulvestad (University of California, San Diego)<br>Operando *Investigation of the Hydriding Phase Transformation in Single Palladium Nanocubes* |
| 2:55 – 3:25 | Break |
| 3:25 – 3:55 | Robert Leheny (Johns Hopkins University)<br>*Connecting Nanoscale Dynamics and Mechanical Properties of Disordered Soft Materials* |
| 3:55 – 4:25 | Reeju Pokharel (Los Alamos National Laboratory)<br>*Utilizing High-energy X-rays for Microstructural Characterization of Nuclear Fuel Materials* |
| 4:25 – 4:55 | David Vine (Argonne National Laboratory)<br>*Ptychography 2025* |
| 4:55 | Adjourn |
| 5:15 | Buses leave APS and Guest House for the banquet at 5:15 sharp! |
| 6:15 | Banquet |

## Parallel Facility Plenary Sessions—Afternoon

## CNM Session
## Bldg. 402, Room E1100-1200

| | |
|---|---|
| **Session Chair:** | **Seungbum Hong (MSD, Argonne National Laboratory) and Steve Smith (NANO SE and South Dakota School of Mines and Technology)** |
| 1:30 – 2:15 | Keynote Speaker<br>Vlad Shalaev (Purdue University, The Robert and Anne Burnett Professor of Electrical and Computer Engineering)<br>*New Material Platform for Plasmonics* |
| 2:15 – 2:45 | Teri Odom (Northwestern University, Board of Lady Managers of the Columbian Exposition Professor of Chemistry, Executive Editor, ACS Photonics)<br>*Room-temperature Lasing from Nanoparticle Arrays* |
| 2:45 – 3:15 | Heinrich Jaeger (The University of Chicago, William J. Friedman and Alicia Townsend Professor of Physics)<br>*Ion Transport Controlled by Nanoparticle-functionalized Membranes* |
| 3:15 – 3:35 | Break |
| 3:35 – 3:45 | Update from CNM Users Executive Committee |
| 3:45 – 4:15 | Alex Martinson (Argonne National Laboratory, Materials Science Division)<br>*Photoexcited Carrier Dynamics in $Cu_2S$ Thin Films* |
| 4:15 – 4:45 | Jonathan J. Foley, IV (Argonne National Laboratory, Nanoscience & Technology Division)<br>*Looking Out for The Tiniest Lights: Controlling Light with Plasmons Across Metal Interfaces* |
| 4:45 – 5:00 | Invited student talk: Vuk Brajuskovic (Northwestern University)<br>*Origins of Anisotropy in the Magnetic Structure of Artificial Spin Ice Lattices* |
| 5:00 | Adjourn |
| 5:15 | Buses leave APS and Guest House for the banquet at 5:15 sharp! |
| 6:15 | Banquet |

# Tuesday, May 12

8:00 – 5:00 Exhibits
*Bldg. 402, Gallery (lower level), outside E1100/1200 and Bldg. 402, Atrium*

8:00 – 5:00 Registration
*Bldg. 402, Atrium*

12:00 – 2:00 Poster setup
*(shuttle buses and vans provided throughout the lunch hour to provide transportation between APS, the Guest House, and TCS Bldg. 240)*

12:00 – 1:30 Lunch
*Tents outside lower level Gallery*

12:00 – 1:30 CNM Users Executive Committee Meeting
*Bldg. 401, Room B5100*

12:00 – 1:30 APS Partner User Council Meeting
*Bldg. 401, Fifth Floor Gallery*

5:00 Dynamic Compression Tour – (Preregistration required)
*Sector 35*

5:30 – 8:00 Poster Session
*TCS Building 240*

## Parallel Facility-specific Workshops*

**CNM** – Workshop 1 (full day) – Bldg. 401, Room A5000
*Nanophotonic Structures, Surfaces, and Composites for Radiative Control (see page 17)*

**APS** – Workshop 2 (full day) – Bldg. 402, Lecture Hall
*High-energy-resolution Inelastic X-ray Scattering (see page 22)*

**APS** – Workshop 3 (full day) – Bldg. 401, Room A1100
*Developing Synchrotron Sample Environments to Study Next-generation Field-driven Device Physics (see page 28)*

**CNM** – Workshop 4 (full day) – Bldg. 402, Room E1100/E1200
*Exploring the Flatland of 2D Materials for Tribological Manipulation (see page 37)*

*Workshop 5 was withdrawn.

## Wednesday, May 13

8:00 – 5:00 Exhibits
*Bldg. 402 Gallery, outside E1100/1200 and Bldg. 402 Atrium*

8:00 – 12:00 Registration
*Bldg. 402, Atrium*

12:00 – 1:30 Lunch
*Tents outside lower level Gallery*

12:00 – 1:30 APSUO Steering Committee Meeting
*Bldg. 401, Fifth Floor Gallery*

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### Parallel Facility-specific Workshops

**APS** – Workshop 3 continued (morning) – Bldg. 432, Conference Room C010
*Developing Synchrotron Sample Environments to Study Next-generation Field-driven Device Physics* *(see page 28)*

**APS** – Workshop 6 (morning) – Bldg. 402, Lecture Hall
*APS Upgrade: Planning the First Experiments* *(see page 43)*

**APS/CNM** – Workshop 7 (full day) – Bldg. 401, Room A1100
*Experimental and Computational Challenges of* in situ *Multimodal Imaging of Energy Materials* *(see page 45)*

**APS** – Workshop 8 (morning) – Bldg. 401, Room A5000
In situ *X-ray Characterization of Microstructure during Manufacture* *(see page 52)*

**APS** – Workshop 9 (morning) – Bldg. 402, Room E1100/E1200
*Workshop on Sn-119 Nuclear Resonant Scattering at the APS* *(see page 55)*

**APS** – Workshop 10 (afternoon) – Bldg. 401, Room A5000
*Application of Synchrotron X-ray Scattering Techniques to Nuclear Materials* *(see page 59)*

**APS** – Workshop 11 (afternoon) – Bldg. 402, Room E1100/E1200
*Tracking Electronic and Structural Dynamics in Proteins and Materials at Sector 14* *(see page 62)*

# Thursday, May 14

## CNM Short Courses (Preregistration is required)

| | |
|---|---|
| 8:30 – 12:00<br>1:30 – 5:00 | Course A: Introduction to Transmission, Scanning Transmission, and Analytical Electron Microscopy<br>*Bldg. 212, Room A157* |
| 8:30 – 12:00 | Course B: Introduction to Confocal Raman Microscopy<br>*Bldg. 440, Main Lobby* |
| 8:30 – 12:00 | Course C: Using the Hard X-ray Nanoprobe<br>*Bldg. 440, Main Lobby* |
| 1:30 – 5:00 | Course D: Atomic Layer Deposition: Theory and Novel Applications<br>*Bldg. 440, Main Lobby* |

# 2015
# APS/CNM Users Meeting

# GENERAL SESSION ABSTRACTS

APS

## Evolving Rock Structure

**Wen-lu Zhu**

**Department of Geology, University of Maryland, College Park, MD 20782**

Correlating physical properties with rock structure at various scales, in three dimensions (3-D), and in four dimensions (4-D, when time evolution is considered), hold the key to unraveling complex geological processes. Synchrotron x-ray imaging, coupled with digital rock physics, is transforming the field of experimental rock deformation by revealing the details of evolving rock microstructures.

3-D images obtained by synchrotron x-ray tomography at nano- and micro-meter scale depict the pore structure of rocks in unprecendented detail. Based on the 3-D microstructure, we determine the bulk electrical conductivity and permeability of partially molten mantle rocks. Numerical experiments of electric currents and fluid flow conducted on the same rock show that the respective pathways for direct current and fluid flow may differ considerably, with more tortuous fluid flow paths in general over the same pore geometry. These results enable better-guided interpretations of geophysical data and constrain melt connectivity and transport at mid-ocean ridges.

Taking advantages of fast developing 4-D x-ray imaging techniques, we investigate the real-time evolution of microscopic pore structure in olivine aggregates reacting with a carbon-rich fluid. The transformation of olivine to magnesium carbonates increases the volume of solids. Our data reveal that after the initial filling of pore space by the growth of new minerals, cracks in polygonal patterns emerge. Subsequently these cracks coalesce into large fractures, which leads to a complete disintegration of the sample. These results provide direct experimental evidence of reaction-induced cracks during mineral carbonation. These data can be used to determine if the reaction between magnesium-iron silicates and carbon-rich fluid is self-sustaining, an important step towards evaluating whether carbon sequestration in ultramafic rocks is feasible.

APS

## *Operando* Investigation of the Hydriding Phase Transformation in Single Palladium Nanocubes

**Andrew Ulvestad[1], Ross Harder[2], Oleg Shpyrko[1], and Paul Mulvaney[3]**

[1]Department of Physics, University of California-San Diego, La Jolla, CA 92093
[2]Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439
[3]School of Chemistry and Bio21 Institute, University of Melbourne, Parkville, VIC 3010, Australia

Phase transitions in reactive environments are crucially important in energy and information storage, catalysis, and sensors. Nanostructuring materials used in these systems can cause a host of desirable properties, including faster charging/discharging kinetics, increased lifespan, and record activities. However, establishing the causal link between structure and function is challenging for nanoparticles as ensemble measurements convolve intrinsic single particle properties with sample size and shape diversity. Here we study the hydriding phase transformation in individual palladium nanocubes under operando conditions using coherent x-ray diffractive imaging. We directly observe two-phase coexistence in the single particle diffraction data. The phase transformation initiates at the corner of the cube, penetrates further into the particle, and eventually violently rearranges the crystal structure. The strain distributions of the $\alpha$ and $\beta$ phases are markedly different, indicating more than a simple Wulff geometric construction is required. A phase field model is constructed to interpret the phase transformation. Our results provide a general framework for understanding phase transformations in individual nanocrystals under operating conditions in reactive environments while highlighting the utility and importance of single particle investigations to truly understand important systems.

APS

## Connecting Nanoscale Dynamics and Mechanical Properties of Disordered Soft Materials

**Robert L. Leheny**

**Department of Physics and Astronomy, Johns Hopkins University, Baltimore, MD 21218**

Complex fluids, such as polymer solutions and colloidal suspensions, are distinguished by their complicated response to mechanical stress. The distinct deformation and flow behaviors of such systems arise from the relaxation of internal structure on the nanometer or micrometer scale; however, the correspondence between this macroscopic mechanical behavior and the microscopic structural dynamics is rarely straightforward. In recent years x-ray photon correlation spectroscopy (XPCS) has emerged as a technique exceptionally suited to characterize the structural dynamics of many complex fluids at the nanometer length scales relevant to their mechanical behavior. This talk will describe a set of related projects employing XPCS on model systems including entangled polymer solutions and colloidal gels that reveal new insight into the microscopic origins of their mechanical properties.

APS

## Utilizing High-energy X-rays for Microstructural Characterization of Nuclear Fuel Materials

**Reeju Pokharel and Donald W. Brown**

**Los Alamos National Laboratory, Los Alamos, NM 87545**

Efforts are underway to develop fundamental understanding of microstructural evolution of nuclear fuel materials, which operate at high temperatures for extended periods of time. High-energy synchrotron x-rays can be utilized to non-destructively probe bulk samples, which enables the study of the microstructural evolution and dynamics of grain growth process in nuclear fuels. Near-field high-energy x-ray diffraction microscopy (nf-HEDM) in conjunction with micro-tomography has provided a unique platform for effective and efficient materials characterization, allowing for spatially resolved crystallographic orientation and density maps measurements in three-dimension. To date, these techniques have been used on metals with relatively low Z number (Cu, Zr, Al, Ni), and have been recently extended to measure ceramic fuels with high Z number, materials which require higher incident photon energy x-rays than previously used. The evolution of different microstructural parameters is monitored to understand the effect of fabrication process on fuel performance as well as microstructural changes driven by the thermal gradient that develops during service conditions. The non-destructive nature of this characterization technique allows for the monitoring of various material processes before, during, and after evolution in extreme environments, and will provide unprecedented data for the development and validation of theoretical codes aimed at predicting fuel performance.

APS

## Ptychography 2025

**D.J. Vine**

**Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439**

Ptychography combined with x-ray fluorescence is a powerful and unique tool for nanoscale imaging. In this presentation, I will speak to how developments over the next decade in source brilliance, scanning microscope design, fast frame rate detectors, and parallel, scalable computational power will revolutionize ptychography. Current and planned projects at the APS have us well placed to take the lead in bringing these advances to bear on exciting scientific problems with national importance.

CNM

## New Material Platform for Plasmonics

**Vladimir M. Shalaev**

Purdue University, West Lafayette, IN 47907

We outline the recent progress in developing new plasmonic materials that will form the basis for future low-loss, CMOS-compatible devices that could enable full-scale development of the metamaterial and nanophotonic technologies.

CNM

## Room-temperature Lasing from Nanoparticle Arrays

**Teri Odom**

Northwestern University, Evanston, IL 60208

This talk will discuss lasing action from band-edge lattice plasmons in arrays of plasmonic nanocavities in a homogeneous dielectric environment. Optically pumped, 2D arrays of plasmonic (Au, Ag) nanoparticles surrounded by an organic gain medium can show directional beam emission (divergence angle < 1.5° and linewidth < 1.3 nm) characteristic of lasing action in the far-field. Lasing in such hybrid systems can be achieved from stimulated energy transfer from the gain to the band-edge lattice plasmons in the deep subwavelength vicinity of individual nanoparticles. Besides our semi-quantum model to explain the lasing action, the idea of a lasing spaser can also be used to describe our system and other array-based cavities. Finally, we will discuss how the emission from lattice-plasmon nanolasers can be dynamically tuned by changing the dielectric environment of the gain media while keeping the nanoparticle-array cavity fixed.

CNM

## Ion Transport Controlled by Nanoparticle-functionalized Membranes

**Edward Barry[1], Sean P. McBride[2], Heinrich M. Jaeger[2,3], and Xiao-Min Lin[1]**

[1]Center for Nanoscale Materials, Argonne National Laboratory, Argonne, IL 60439
[2]James Franck Institute, University of Chicago, Chicago, IL 60637
[3]Department of Physics, University of Chicago, Chicago, IL 60637

From proton exchange membranes in fuel cells to ion channels in biological membranes, the well-specified control of ionic interactions in confined geometries profoundly influences the transport and selectivity of porous materials. Here we outline a versatile new approach to control a membrane's electrostatic interactions with ions by depositing ligand-coated nanoparticles around the pore entrances. Leveraging the flexibility and control by which ligated nanoparticles can be synthesized, we demonstrate how ligand terminal groups such as methyl, carboxyl, and amine can be used to tune the membrane charge density and control ion transport. Further functionality, exploiting the ligands as binding sites, is demonstrated for sulfonate groups resulting in an enhancement of the membrane charge density. We then extend these results to smaller dimensions by systematically varying the underlying pore diameter. As a whole, these results outline a previously unexplored method for the nanoparticle-functionalization of membranes using ligated nanoparticles to control ion transport.

CNM

## Photoexcited Carrier Dynamics of $Cu_2S$ Thin Films for Photovoltaics

**Shannon C. Riha[1], Richard D. Schaller[2], David J. Gosztola[2], Gary P. Wiederrecht[2], and Alex B.F. Martinson[1]**

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Copper sulfide is a simple binary material with promising attributes for low-cost thin film photovoltaics. However, stable $Cu_2S$-based device efficiencies approaching 10% free from cadmium have yet to be realized. We utilize transient absorption spectroscopy at the CNM to investigate the dynamics of the photoexcited state of isolated $Cu_2S$ thin films

prepared by atomic layer deposition (ALD) or vapor-based cation exchange of ZnS. While a number of variables including film thickness, carrier concentration, surface oxidation, and grain boundary passivation were examined, grain structure alone was found to correlate with longer lifetimes. A map of excited state dynamics is deduced from the spectral evolution from 300 fs to 300 μs. This study provides insights into why such high device efficiencies may have been achieved in CdS/$Cu_2S$ hetero-structure devices fabricated through topotaxial exchange processes. Revealing the effects of grain morphology on the photophysical properties of $Cu_2S$ is a crucial step toward reaching high efficiencies in operationally stable $Cu_2S$ thin film photovoltaics.

CNM

## Looking Out for the Tiniest Lights: Controlling Light with Heterogeneous Interfaces

**Jay Foley[1], Hayk Harutyunyan[1,2], Daniel Rosenmann[1], Zheng Li[1], Sheng Peng[1] , Jeff McMahon[3], George Schatz[3], Gary Wiederrecht[1], Yugang Sun[1], and Stephen Gray[1]**

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Research in nanophotonics, which seeks to understand and control optical energy and information flow at nanoscale dimensions, has been motivated by a number of applications ranging from energy conversion, imaging, and information storage, to name a few. The ability to confine optical energy beyond the diffraction limit by coupling light into collective electronic motion in metal nanostructures, including localized surface plasmons on metal nanoparticles and surface plasmon polaritons on metal surfaces, has played a key role in many advances in nanophotonics. Consequently, discovering and understanding plasmonics phenomena in new contexts is a considerable focus of nanophotonics research.

This talk will focus on plasmonics phenomena in the context of bimetallic interfaces. I'll describe an entirely new type of plasmon known as an "Inhomogeneous Surface Plasmon Polariton" (ISPP). ISPPs provide new opportunities for controlling the propagation behavior and confinement of light, and can be generated by refraction of ordinary surface plasmon polaritons at the interface between two metal surfaces. I will also describe the enhanced plasmonic properties of interfaced bimetallic nanoparticles, including their ability to support so-called charge-transfer plasmons.

CNM

## Origins of Anisotropy in the Magnetic Structure of Artificial Spin Ice Lattices

**Vuk Brajuskovic[1,2], Charudatta Phatak[1], and Amanda Petford-Long[1,2]**

[1]Materials Science Division, Argonne National Laboratory, Argonne, IL 60439
[2]Department of Materials Science and Engineering, Northwestern University, Evanston, IL 60208

Artificial spin ice lattices are a two-dimensional analogue to spin ice crystals. The larger length scales of the artificial systems allow for their magnetic frustration behavior to be observed at room temperature. Traditionally, artificial spin ice lattices are patterned in thin films using electron-beam lithography because the elements that make up the lattice need to be small if they are to remain single domain. However, such fabrication is difficult to perform when the thin films have been deposited onto transparent membranes on TEM grids. Therefore, for those lattices whose elements remain single-domain at larger sizes, focused-ion beam (FIB) patterning represents a better alternative. In order to explore the origins of observed anisotropy in the lattices, Lorentz transmission electron microscopy has been used to obtain quantitative maps of the magnetization in square spin ice lattices FIB patterned in $Ni_{80}Fe_{20}$ (Permalloy) thin films. Our results have shown that although FIB patterning has an effect on the grain size of the Permalloy at the edges of the patterned regions, the anisotropy that is observed is the result of pre-existing anisotropy in the Permalloy film, rather being than an effect introduced by the FIB patterning. Our results thus suggest that FIB patterning is an effective method for fabricating larger-scale artificial spin ice lattices.

*This work was supported by the U.S. Department of Energy, Office of Basic Energy Sciences, Division of Material Sciences and Engineering. Use of the Center for Nanoscale Materials was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.*

# 2015

# APS/CNM Users Meeting

# WORKSHOP AGENDAS AND ABSTRACTS

# Tuesday, May 12
# Facility-specific Workshops

## CNM Workshop 1
## Nanophotonic Structures, Surfaces, and Composites for Radiative Control

Location: Bldg. 401, Room A5000

Organizers: Steve Smith (South Dakota School of Mines & Technology), Il Woong Jung (Nanofabrication & Devices Group, CNM), Zhe Fei (Nanophotonics Group, CNM))

The scope of this workshop encompasses all aspects of the photonic control of quantum systems, including the fabrication, characterization, simulation, and theory of composite photonic-electronic systems, that is, those specific materials and nano-structured composite systems which intentionally modify and/or exhibit control of light matter interactions as a consequence of their nanostructure. Examples are quantum emitters (e.g., molecules, ions or quantum dots) whose radiative properties are modified by their interactions with nano-optical structures, including photonic crystals, cavities, plasmonic surfaces and/or high-aspect ratio optical nano-structures. Development of improved optical materials, nanostructures, and composites and their applications will be emphasized as well as photonic crystals, micro-cavities, photonic control, plasmonics, fabrication, and the properties of photonic nano-structures.

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| 8:30 – 8:45 | Welcome & Introductory Remarks |
| 8:45 – 9:25 | Marko Loncar (Harvard University)<br>*Quantum and Nonlinear Nanophotonics with Diamond* |
| 9:25 – 10:05 | George Schatz (Northwestern University)<br>*Plasmonic Arrays* |
| 10:05 – 10:35 | Break |
| 10:35 – 11:15 | Yugang Sun (Argonne National Laboratory)<br>*Interfacial Influence on Surface Plasmon Resonances in "Quantum-sized" Metal Nanoparticles* |
| 11:15 – 12:00 | Zhiqun Lin (Georgia Tech)<br>*Crafting Nanophotonic Structures* |
| 12:00 – 1:30 | Lunch |
| 1:30 – 2:10 | Peng Zhang (University of Cincinnati)<br>*SERS Inside Metal Nanoshell and Its Applications* |
| 2:10 – 2:50 | Shaung Fang Lim (North Carolina State University)<br>*Multifunctional Diagnostic, Nanothermometer and Photothermal Nano-devices* |
| 2:50 – 3:20 | Break |
| 3:20 – 4:00 | Stephen Gray (Argonne National Laboratory)<br>*Theoretical Studies of Hybrid Plasmon/Quantum-dot Systems* |

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| 4:00 – 4:40 | Nathaniel Stern (Northwestern University)<br>*Photons in Flatland: Manipulating Light and Matter in Two-dimensional Nanomaterials* |
| 4:40 | Wrap-up and concluding remarks |

WK1

## Quantum and Nonlinear Nanophotonics with Diamond

**Marko Loncar**

**School of Engineering and Applied Science, Harvard University, Cambridge, MA 02138**

Diamond possesses remarkable physical and chemical properties, and in many ways is the ultimate engineering material — “the engineer’s best friend!” For example, it has high mechanical hardness and large Young’s modulus, and is one of the best thermal conductors. Optically, diamond is transparent from the ultra-violet to infra-red, has a high refractive index (n = 2.4), strong optical nonlinearity and a wide variety of light-emitting defects. Finally, it is biocompatible and chemically inert, suitable for operation in harsh environment. These properties make diamond a highly desirable material for many applications, including high-frequency micro- and nano-electromechanical systems, nonlinear optics, magnetic and electric field sensing, biomedicine, and oil discovery. One particularly exciting application of diamond is in the field of quantum information science and technology, which promises realization of powerful quantum computers capable of tackling problems that cannot be solved using classical approaches, as well as realization of secure communication channels. At the heart of these applications are diamond’s luminescent defects — color centers — and the nitrogen-vacancy (NV) and silicon-vacancy (SiV) color center in particular. These atomic systems in the solid-state possesses all the essential elements for quantum technology, including storage, logic, and communication of quantum information.

I will review recent advances in nanotechnology that have enabled fabrication of nanoscale optical devices and chip-scale systems in diamond that can generate, manipulate, and store optical signals at the single-photon level. Examples include a room temperature source of single photons based on diamond nanowires [1] and plasmonic appertures [2], as well as single-photon generation and routing inside ring [3] and photonic crystal resonators [4]. Novel, fabrication technique [5] — *angled-etching* — suitable for realization of nanophotonic [6] and nanomechanic [7,8] devices in bulk diamond crystals will also be discussed. Finally, I will present our recent result on frequency comb generation at telecom wavelengths using high Q factor ($>10^6$) diamond ring resonator [9], and discuss our ongoing efforts towards realization of diamond comb in visible range. (http://nano-optics.seas.harvard.edu.)

*This work has been supported by DARPA, AFOSR and NSF.*

[1] T.M. Babinec et al., “A bright single photon source based on a diamond nanowire,” *Nature Nanotechnology* **5**, 195 (2010).

[2] J.T. Choy et al., “Enhanced Single Photon Emission by Diamond-Plasmon Nanostructures,” *Nature Photonics* **5**, 738 (2011).

[3] B.J.M. Hausmann et al., “Integrated Diamond Networks for Quantum Nanophotonics,” *Nano Letters* **12**, 1578 (2012).

[4] B.J.M. Hausmann et al., “Coupling of NV centers to photonic crystal nanobeams in diamond,” *Nano Letters* **13**, 5791–5796 (2013).

[5] M.J. Burek et al., “Free-standing mechanical and photonic nanostructures in single-crystal diamond,” *Nano Lett.* **12**, 6084 (2012).

[6] M. Burek, Y. Chu, M. Liddy, P. Patel, J. Rochman, W. Hong, Q. Quan, M.D. Lukin, and M. Loncar, “High-Q optical nanocavities in bulk single-crystal diamond,” *Nat. Comm.* **5**, 5718 (2014).

[7] M. Burek et al., “Nanomechanical resonant structures in single-crystal diamond,” *Appl. Phys. Lett.* **103**, 131904 (2013).

[8] Y.I. Sohn et al., “Dynamic Actuation of Single-Crystal Diamond Nanobeams,” *arXiv:1408.5822.*

[9] B. Hausmann et al., “Diamond Nonlinear Photonics,” *Nature Photonics* **8**, 369 (2014).

WK1

## Plasmonic Arrays

**George C. Schatz**
Northwestern University, Evanston, IL 60208

Silver and gold nanoparticles have unique optical properties that are associated with the excitation of collective excitations of the conduction electrons known as plasmon resonances. The resonance frequencies are sensitive to particle shape and size, which means that the color of the nanoparticles can be tuned over a wide range of wavelengths, and they are also sensitive to the arrangement of the nanoparticles into aggregates and arrays. This talk will emphasize recent theory and experiments that have probed the effect of arrays of these particles in 1D, 2D and 3D on optical response. The arrays in 1D and 2D can be made using standard lithography tools, but much of the talk will emphasize bottom-up assembly of arrays that is possible using DNA-functionalized nanoparticles and self-assembly of nanoparticle superlattices driven by DNA hybridization. We show that the array structures lead to new kinds of hybrid optical modes in which localized surface plasmon resonances in the nanoparticles are coupled with photonic modes of the lattices, including Bragg modes, Fabry-Perot modes and other modes. These hybrid modes are often much narrower than the isolated particle plasmons, and films composed of these superlattices have unusual metamaterials properties. We also show that for 2D lattices it possible to generate a new class of sub-wavelength laser in which excitons in laser dyes are coupled with the hybrid lattice modes to produce enhanced stimulated emission.

WK1

## Interfacial Influence on Surface Plasmon Resonances in “Quantum-sized” Metal Nanoparticles

**Sheng Peng, Zheng Li, Jonathan J. Foley, IV, Stephen K. Gray, Gary P. Wiederrecht, and Yugang Sun**
Center for Nanoscale Materials, Argonne National Laboratory, Argonne, IL 60439

Nanoparticles made of noble metals such as silver and gold exhibit strong optical absorption due to their surface plasmon resonance (SPR) that corresponds to the collective oscillation of surface conduction electrons in response to the incident electromagnetic waves. As the nanoparticles are smaller than 20 nm (i.e., the size in the range similar to that of quantum dots), the interfaces formed on the surfaces of these noble metal nanoparticles significantly influence the nanoparticles’ SPR. For example, the monodispersed silver nanoparticles synthesized *via* a well-defined chemical reduction process exhibit an exceptional size-dependence of SPR peak positions: as particle size decreases from 20 nm the peaks blue-shifts but then turns over near ~12 nm and strongly red-shifts. Theoretic modeling and calculations reveal that the surface chemistry corresponding to the interactions between the capping molecules and the surface silver atoms in the nanoparticles become pronounced in determining their optical properties because the surface silver atoms represent a significant fraction of the total number of atoms in small nanoparticles. Such surface chemistry reduces the density of conduction band electrons (i.e., free electrons) in the surface layer of metal atoms, thus consequently influences the frequency-dependent dielectric constant of the metal atoms in the surface layer and the overall SPR absorption spectrum. In this presentation, a number of interfaces including the aforementioned metal/surfactant interface, metal/inorganic dielectric interface, and metal/metal interface will be discussed to highlight the importance of interface conditions on SPRs in “quantum-sized” noble metal nanoparticles. A comprehensive understanding of the relationship between interfacial coupling/chemistry and optical properties will be beneficial to exploit new applications of small colloidal metal nanoparticles, such as colorimetric sensing, electrochromic devices, surface enhanced spectroscopies, and photocatalysis.

*This work was performed at the Center for Nanoscale Materials, a U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences User Facility under Contract No. DE-AC02-06CH11357.*

WK1

## Crafting Nanophotonic Structures

**Zhiqun Lin**
**School of Materials Science and Engineering, Georgia Institute of Technology, Atlanta, GA 30332**

Colloidal nanocrystals exhibit a wide range of size and shape dependent properties and have found application in a myriad of fields such as photonics, optics, electronics, mechanics, drug delivery and catalysis to name but a few. Synthetic protocols that enable simple and convenient production of colloidal nanocrystals with controlled size, shape and composition are therefore of key general importance. Current strategies, however, often require stringent experimental conditions, are difficult to generalize, or require tedious multi-step reactions and purification. Recently, linear amphiphilic block copolymer micelles have been used as template for the synthesis of functional nanocrystals, but the thermodynamic instability of these micelles limits the scope of this approach. In this talk, I will elaborate general strategies for synthesizing a large variety of functional nanoparticles and nanorods (in particular, photonic nanoparticles and nanorods) with precisely controlled dimensions, compositions and architectures by using nonlinear block copolymers as templates. This new class of copolymers forms unimolecular micelles that are structurally stable under various experimental conditions and therefore overcomes the intrinsic instability of linear block copolymer micelles. Our approach enables the facile synthesis of organic solvent- and water-soluble nearly monodisperse photonic nanoparticles and nanorods with desired composition and architecture, including core/shell and hollow nanostructures.

WK1

## SERS Inside Metal Nanoshell and Its Applications

**Peng Zhang**
**Department of Chemistry, University of Cincinnati, Cincinnati, OH 45221**

The observation of giant enhancement of Surface-enhanced Raman scattering (SERS) in the late 1990s had reignited the tremendous interests in the field. A great variety of nanostructures have been explored as SERS substrates, with various successes in terms of consistency and reproducibility. We approached the subject from a different aspect by placing the Raman probe molecules inside the metal nanoshell, in contrast to other previous experimental works, where SERS effect was studied only for molecules located in the proximal distance outside a nanostructure.

We show that enormous SERS enhancement inside metal (Au, Ag or mixed Ag/Au) nanoshell can be consistently and reproducibly achieved experimentally in the solution. The results provide new insights to the SERS effect, and pave ways for a new design of nanostructures to be used in Raman-based assays and imaging. Nanostructures of such design may also have profound implication in other plasmonic applications.

WK1

## Multifunctional Diagnostic, Nanothermometer, and Photothermal Nano-devices

**Kory Green, Janina Wirth, Megan O'Connor, and Shuang Fang Lim**
**Department of Physics, North Carolina State University, Raleigh, NC 27595**

In this study, the known therapeutic capabilities of gold nanorods (AuNRs) have been combined with the diagnostic and nanothermometer abilities of upconversion nanoparticles (UCNPs) to develop a system for simultaneous biological imaging, photothermal therapy, and nanothermal sensing. Both the excitation of UCNPs and the finely tuned longitudinal surface plasmon resonance (LSPR) mode of AuNRs lay in a window of relatively high light penetration of tissue in the infrared. The nanothermometer property of the UCNPs allows direct quantification of the localized temperature of the photothermally heated AuNRs chemically adsorbed to their surface and is free from the bleaching problems inherent in dye thermal sensing systems, especially at high laser fluences required to kill tissue. Spectroscopy on single particles, verified by transmission electron microscopy (TEM), has been performed at varying temperatures to confirm 1) the thermal sensing properties of UCNPs and 2) to finely tune their upconversion enhancement arising from the LSPR coupling of the AuNRs. Preliminary quantification of the localized AuNR temperatures upon photothermal heating will be confirmed through single particle spectroscopy of the attached UCNPs. HeLa cell viability studies have also been performed.

WK1

## Theoretical Studies of Hybrid Plasmon/Quantum-dot Systems

**Stephen K. Gray**

Center for Nanoscale Materials, Argonne National Laboratory Argonne, IL 60439

I discuss classical electrodynamics and cavity quantum electrodynamics approaches to understanding and predicting the behavior of systems composed of metal nanoparticles and quantum dots such as CdSe nanocrystals. The plasmon resonance of the metal nanoparticle can significantly alter the radiative properties of the quantum dots and its correct incorporation into the modeling is emphasized. New avenues in nonlinear optical phenomena and quantum information are explored with these systems.

WK1

## Photons in Flatland: Manipulating Light and Matter in Two-dimensional Nanomaterials

**Nathaniel P. Stern**

Department of Physics and Astronomy, Northwestern University, Evanston, IL 60208

Hybrid systems integrating light with matter offer a highly controllable landscape for understanding the interface between disparate physical entities. The emergence of materials with atomic-scale thickness suggests a new landscape in which to play with the coupling between light and low-dimensional materials. Exemplifying the interest of this new regime, the crystal symmetry of monolayer two-dimensional (2D) semiconductors can exhibit degenerate, yet distinct, valleys in momentum space that can be separately addressed by polarized light. In this talk, I will describe how these optical transitions in 2D semiconductors can be harnessed for manipulating electronic excitations and charge motion in two-dimensional materials. Drawing on the developments in cavity quantum electrodynamics of the last several decades, I will discuss the behavior of 2D semiconductors when integrated into optical cavities and photonic devices. I will use this approach to illustrate the potential for exploring new hybrid regimes of light-matter coupling based on engineering quantum interactions with nanoscale materials.

# Tuesday, May 12
# Facility-specific Workshops

## APS Workshop 2
## High-energy-resolution Inelastic X-ray Scattering

Location: Bldg. 401, Lecture Hall

Organizers: Ayman Said, Ahmet Alatas, and Bogdan M. Leu (APS)

The workshop is intended as a forum for researchers using high-energy-resolution inelastic x-ray scattering (IXS) to present recent results. Diverse areas of research, including superconductivity, geophysics, charge density waves, and quantum phase transitions, will be covered, as well as theoretical approaches to IXS. The timing of this workshop is ideal for us to update users on recent developments that took place on the HERIX instruments and to discuss new ideas.

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| 8:30 – 8:35 | Linda Young (Argonne National Laboratory)<br>*Welcome & Introductory Remarks* |
| 8:35 – 8:45 | Ayman Said (Argonne National Laboratory)<br>*An Update about Sector 30* |
| 8:45 – 9:25 | Frank Weber (Karlsruhe Institute of Technology)<br>*Soft Phonon Mode and Wave Vector Dependent Electron-phonon Coupling at Charge-density Wave* |
| 9:25 – 10:05 | Matthieu Le Tacon (Max Planck Institute for Solid State Research)<br>*CDW and Electron-phonon Interaction in High-temperature Superconducting Cuprates* |
| 10:05 – 10:35 | Break |
| 10:35 – 11:15 | Jiawang Hong (Oak Ridge National Laboratory)<br>*Modeling the Phonon Dynamical Structure Factor S(Q,E) from First-principles Calculations: The Case Study of Thermoelectrics and Complex Oxides* |
| 11:15 – 12:00 | Charles W. Myles<br>*Clathrate Semiconductors: Novel, Open Framework, Crystalline Materials Based on Si, Ge, and Sn* |
| 12:00 – 1:30 | Lunch |
| 1:30 – 2:10 | John Budai (Oak Ridge National Laboratory)<br>*Comprehensive Scattering Studies of Microstructure, Anharmonic Phonons and Thermodynamics near the Metal-insulator Transition in* $VO_2$ |
| 2:10 – 2:50 | Jason Hancock (University of Connecticut)<br>*Critical Soft Modes and Negative Thermal Expansion* |
| 2:50 – 3:20 | Break |

3:20 – 4:00 Jung-Fu Lin (The University of Texas at Austin)
*High-pressure Acoustic Phonons and Elasticity of Iron Alloys and Oxides*

4:00 – 4:40 Nicholas Butch (NIST Center for Neutron Research)
*Looking for Hidden Phonons in $URu_2Si_2$*

4:40 – 5:00 Wrap-up and concluding remarks

5:00 Adjourn

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WK2

## Soft Phonon Mode and Wave Vector Dependent Electron-phonon Coupling at Charge-density-wave Transitions

**Frank Weber[1], Michael Maschek[1], Stephan Rosenkranz[2], Rolf Heid[1], Roland Hott[1], Dmitry Reznik[3], Goran Karapetrov[4], P. Giraldo-Gallo[5,7], I.R. Fisher[6,7], A. Alatas[8], and Ayman H. Said[8]**

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[2] Materials Science Division, Argonne National Laboratory, Argonne, IL 60439
[3] Department of Physics, University of Colorado at Boulder, Boulder, CO 80309
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[5] Geballe Laboratory for Advanced Physics and Department of Physics, Stanford University, CA 94305
[6] Geballe Laboratory for Advanced Physics and Department of Applied Physics, Stanford University, CA 94305
[7] The Stanford Institute for Materials and Energy Sciences, SLAC National Accelerator Laboratory, Menlo Park, CA 94025
[8] Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439

I will review our work on lattice dynamics in the charge-density-wave (CDW) compounds *2H*-$NbSe_2$ [1], *1T*-$TiSe_2$ [2] and $TbTe_3$ [3]. Each of them is different with regard to the interplay of electronic structure and electron-phonon coupling properties. Nevertheless, all emphasize the general applicability of the stability criterion derived for a CDW by Chan & Heine [4].

$$\frac{4\eta_q^2}{\hbar\omega_{bare}} \geq \frac{1}{\chi_q} + \left(2\tilde{U}_q - \tilde{V}_q\right)$$

where $\eta_q$ is the electron-phonon coupling associated with a mode at a bare energy of $\hbar\omega_{bare}$, $\chi_q$ is the dielectric response of the conduction electrons, and $\tilde{U}_q$ and $\tilde{V}_q$ are their Coulomb and exchange interactions. Although both sides of this inequality are essential in stabilizing the CDW order, the common assumption is that the modulation wave vector, $\vec{q}_{CDW}$, is determined by the right-hand side (i.e., by a singularity in the electronic dielectric function χq originating from a Fermi surface nesting).

Using high-energy resolution inelastic x-ray scattering and ab initio calculations of the lattice dynamical properties we analyzed the situation in the three above mentioned materials and find the following:

- There is no Fermi surface nesting in *2H*-$NbSe_2$ and $\boldsymbol{q}_{CDW}$ is completely defined by $\eta_q$.
- $TbTe_3$ features a weak nesting close to $\boldsymbol{q}_{CDW}$ but only including $\eta_q$ yields the correct ordering wave vector.
- $\eta_q$ in *1T*-$TiSe_2$ has a comparatively small wave vector dependence and the **q** dependence of one particular electronic scattering path defines $\boldsymbol{q}_{CDW}$.

[1] F. Weber, S. Rosenkranz, J.P. Castellan, R. Osborn, R. Hott, R. Heid, K.P. Bohnen, T. Egami, A.H. Said, and D. Reznik, *Physical Review Letters* **107** (10), 107403 (2011); F. Weber, R. Hott, R. Heid, K.P. Bohnen, S. Rosenkranz, J.P. Castellan, R. Osborn, A.H. Said, B.M. Leu, and D. Reznik, Physical Review B **87** (24), 245111 (2013).

[2] F. Weber, S. Rosenkranz, J.P. Castellan, R. Osborn, G. Karapetrov, R. Hott, R. Heid, K.P. Bohnen, and A. Alatas, *Physical Review Letters* **107** (26), 266401 (2011).

[3] M. Maschek, S. Rosenkranz, R. Heid, A. Said, P. Giraldo-Gallo, I.R. Fisher, and F. Weber, arXiv: cond-mat:1410.7592 (2014).

[4] S.K. Chan and V. Heine, *Journal of Physics F: Metal Physics* **3**, 795 (1973).

WK2

## CDW and Electron-phonon Interaction in High-temperature Superconducting Cuprates

**Matthieu Le Tacon**

Max Planck Institute for Solid State Research, Stuttgart, 70569, Germany

I will present an overview of the results obtained from various x-ray scattering experiments on high temperature superconducting cuprates in the last couple of years. I will first focus on Cu L-edge resonant scattering experiments that led us to uncover charge density wave (CDW) correlations competing with superconductivity in the YBCO family [1–3], for which a complete temperature and doping dependent phase diagram has been worked out [4]. Further information was gained from high-resolution inelastic x-ray scattering that allows us to discuss the nature of the CDW. The observation of a quasi-elastic 'central peak' unraveled the static nature of the CDW correlations, attributed to the pining of CDW nanodomains on defects. Low energy phonons exhibit anomalously large superconductivity induced renormalizations close to the CDW ordering wave vector, providing new insights regarding the long-standing debate of the role of the electron-phonon interaction in the cuprates, a major factor influencing the competition between collective instabilities in correlated-electron materials [5].

[1] G. Ghiringhelli et al., *Science* **337**, 821 (2012).

[2] A. J. Achkar et al., *Phys. Rev. Lett.* **109**, 167001 (2012).

[3] S. Blanco-Canosa et al., *Phys. Rev. Lett.* **110**, 187001 (2013).

[4] S. Blanco-Canosa et al., *Phys. Rev. B* **90**, 054513 (2014).

[5] M. Le Tacon et al., *Nature Physics* **10**, 52 (2014).

WK2

## Modeling the Phonon Dynamical Structure Factor S(Q,E) from First-principles Calculations: The Case Study of Thermoelectrics and Complex Oxides

**Jiawang Hong, Jennifer Niedziela, Chen Li, Vickie Lynch, and Olivier Delaire**

Oak Ridge National Laboratory, Oak Ridge, TN 37830

SimPhonies, a python-based application developed within CAMM at SNS, enables users to calculate the x-ray/neutron-weighted dynamical structure factor for phonon scattering, S(**Q**,E), from first-principles calculations. It enables direct, quantitative comparison with large datasets from inelastic x-ray/neutron scattering measurements on single-crystals. The SimPhonies code leverages first-principles electronic structure packages to calculate the x-ray/neutron scattering intensities convolved with instrumental resolution, including anharmonic effects, and outputs the scattering in the region of interest in reciprocal space. The benefits of reliable S(**Q**,E) simulations, and comparisons with measurements at HERIX/SNS, are illustrated with several scientific studies in a wide range of materials, including thermoelectrics, ferroelectrics and complex oxides.

*Research was supported by the Center for Accelerating Materials Modeling from SNS data (CAMM), funded by the U.S. DOE, BES, MSED.*

WK2

## Clathrate Semiconductors: Novel, Open Framework, Crystalline Materials Based on Si, Ge, and Sn

**Charles W. Myles and Dong Xue**

**Department of Physics, Texas Tech University, Lubbock, TX 79409**

The Group IV elements Si, Ge, and Sn usually crystallize in the diamond lattice structure, which is the ground state phase for each. Less well-known is the fact that these elements can also form novel crystalline solids, called clathrates because of their structural similarities to clathrate hydrates. Group IV clathrates are metastable, expanded volume phases. As in the diamond structure, in the clathrates, the atoms are tetrahedrally coordinated in $sp^3$ covalent bonding configurations with their near-neighbors. In contrast to the diamond lattice, however, the clathrates contain pentagonal rings of atoms and their lattices are open frameworks containing large (20-, 24-, 28-atom) "cages". The two common clathrate varieties are Type I, a simple cubic lattice with 46 atoms per unit cell and Type II, a face centered cubic lattice with 34 atoms per unit cell. The cages can contain weakly bound impurities ("guests"), usually Group I or Group II atoms. A reason that the clathrates are interesting is that the choice of guest may be used to tune the material properties. The guests act as electron donors, but because of their weak bonding, they have only small effects on the host electronic band structures. However, they can vibrate with low frequency vibrational modes, which can strongly affect the vibrational properties. Some laboratory-synthesized, guest-containing clathrates show promise for thermoelectric applications precisely because the guests only weakly perturb the electronic properties, while strongly affecting the vibrational properties.

In this talk, the clathrates and their lattices will be introduced. The results of calculations of the properties of some Si, Ge and Sn-based Type I and Type II clathrates will then be presented. Where data is available, some results will be compared with experiments. Our calculations are motivated by experiments performed by G. Nolas and his group [1] at the U. of South Florida. Among the experimental techniques they have used to study the structural properties of these materials is powder x-ray diffraction (XRD). Our calculations have used a density-functional based, planewave, pseudopotential method. The results include equations of state, structural parameters, electronic bands, vibrational spectra, mean-square atomic displacements, and thermodynamic properties. Some recent results [2–5], obtained in collaboration with some present and former students will be discussed.

[1] M. Beekman, E.N. Nenghabi, K. Biswas, C. Myles, M. Baitinger, Y. Grin, and G.S. Nolas, *Inorganic Chemistry* **49**, 5338 (2010).

[2] Dong Xue and C. Myles, in preparation. See on-line March, 2015 APS Bulletin, papers F12-00007 and F12-00008.

[3] E.N. Nenghabi and C. Myles, *Phys. Rev.* B **77**, 205203 (2008); *Phys. Rev.* B **78**, 195202 (2008).

[4] K. Biswas, C. Myles, M. Sanati, and G. Nolas, *J. Appl. Phys.* **104**, 033535 (2008).

[5] P. Norouzzadeh, C. Myles, and D. Vashaee, *J. Appl. Phys.* **114**, 163509 (2013); *J. Alloys and Compounds* **587**, 474 (2014).

WK2

## Comprehensive Scattering Studies of Microstructure, Anharmonic Phonons, and Thermodynamics near the Metal-insulator Transition in $VO_2$

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Vanadium dioxide is a strongly correlated material that exhibits a well-studied, but poorly understood, metal-insulator transition (MIT) coupled with a tetragonal (rutile) to monoclinic (M1) structural phase transition just above room temperature. Competing models for this coupled electronic and structural transition are based on either a Peierls MIT driven by instabilities in electron–lattice dynamics or a Mott MIT where strong electron–electron correlations drive charge localization. In spite of early predictions of a soft-mode lattice instability at the rutile R-point, phonon dispersions have not been measured in $VO_2$ because the incoherent neutron scattering cross-section precludes traditional single-crystal neutron scattering measurements. To shed light on changes in microstructure, lattice dynamics, and thermodynamics in $VO_2$, we have combined a comprehensive set of x-ray and neutron scattering results with *ab initio* molecular dynamics calculations (AIMD). Four complementary scattering studies included: (1) x-ray microdiffraction at APS 33-BM, (2) single-crystal inelastic x-ray scattering studies of phonon dispersions at APS-HERIX, (3) inelastic neutron powder measurements of phonon density of states at SNS-ARCS, and (4) x-ray thermal diffuse scattering at APS 33-BM. We found good agreement between the experimental results and the AIMD predictions, and the calculations help reveal the physical mechanisms driving the MIT. Our results show, first, that previous proposals of a "soft mode" phase transition at a particular wavevector are incorrect. Second, we find that changes in the phonon spectra across the transition are responsible for most of the total entropy change, that is, the entropy change is dominated by vibrational rather than electronic contributions. Finally, we observe strongly anharmonic phonons across a wide region of reciprocal space in the tetragonal phase, and conclude that these short-lived, low-energy phonons are responsible for thermodynamically stabilizing the metallic phase at high temperatures.

*Support by DOE Office of Basic Energy Sciences, Materials Sciences and Engineering Division; APS and SNS facilities supported by DOE-BES.*

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## Critical Soft Modes and Negative Thermal Expansion

**Jason Hancock**

**University of Connecticut, Storrs, CT 06269**

We report a high resolution inelastic x-ray scattering study of phonons in a perovskite material which not only displays strong negative thermal expansion, but also exhibits a particularly stable high symmetry cubic phase down to 0.5 K. Our observations include a strong softening of a branch of excitations as the system is cooled, and strong signatures of critical fluctuations are apparent at the lowest temperatures measured. General aspects of the avoided ferroelastic phase boundary to a lower symmetry phase and the broad connection to the physics of perovskite materials will be discussed.

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## High-pressure Acoustic Phonons and Elasticity of Iron Alloys and Oxides

**Jung-Fu Lin**

**Department of Geological Science, The University of Texas at Austin, Austin, TX 78712**

Iron is the most abundant transition metal in the universe and also exists abundantly in the Earth's interior. Studying the physical and chemical properties of iron-bearing compounds, including Fe-Ni-Si alloys and oxides (e.g., magnetite, hematite, wustite), in extreme pressure-temperature environments are thus of great interest to deep-Earth mineral physicists as well as condensed matter physicists [1–3]. Due to the nature of the unfilled *3d* electronic orbitals, these compounds can potentially undergo a number of transitions in extreme conditions including structural, magnetic, spin-pairing, metallic, and/or charge-ordering. These transitions can significantly affect our understanding of the properties of these Fe-bearing compounds at high pressures. Since these compounds are opaque to visible lights, studying their elastic properties had been a challenge. In this presentation, I will highlight the use of the high-energy-resolution inelastic x-ray scattering (HERIX) technique coupled with a high-pressure diamond anvil cell at the Sector 3 and Sector 30 of the Advanced Photon Source to investigate the behavior of acoustic phonons and elasticity (full elastic tensors) of iron alloys and oxides at high pressures and/or temperatures. These experimental elasticity results, together with complimentary x-ray and laser spectroscopic measurements, are used to understand a number of outstanding questions in these systems including how the charge-ordering occurs in the mixed-valence magnetite at high pressure [1] and what are the alloying effects of light elements on the velocity profiles of iron in the deep-Earth core [2,3].

[1] J.F. Lin, J. Wu, J. Zhu, Z. Mao, A. H. Said, B. M. Leu, J. Cheng, Y. Uwatoko, C. Jin¸ and J. Zhou, Abnormal elastic and vibrational behaviors of magnetite at high pressures, *Scientific Reports* **2**, 6282, 2014.

[2] J. Liu, J.F. Lin, A. Alatas, and W. Bi, Sound velocities of bcc-Fe and $Fe_{0.85}Si_{0.15}$ alloy at high pressure and temperature, *Phys. Earth Planet. Inter.* **233**, 24–32, 2014.

[3] Z. Mao, J.F. Lin, J. Liu, A. Alatas, L. Gao, J. Zhao, and H.K. Mao, Sound velocities of Fe and Fe-Si alloys in the Earth's core, *Proc. Natl. Acad. Sci.* **109**, 10239–10244, 2012.

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## Looking for Hidden Phonons in $URu_2Si_2$

**Nicholas Butch**

NIST Center for Neutron Research, Gaithersburg, MD 20899

Among the outstanding problems in condensed matter physics, the identity of the order parameter in the hidden order phase of $URu_2Si_2$ is among the most conspicuous. I will discuss our recent x-ray and neutron study of the lattice, and inevitably, magnetic excitations in this material. I will highlight how HERIX measurements were essential to conclusively disentangle phonon dispersions from the magnetic excitations along certain directions. As a result, we have calculated directly the phonon contribution to the specific heat and analyzed the temperature dependence of the magnetic excitations. I will also present our measurement of phonons under pressure.

# Tuesday, May 12
# Facility-specific Workshops

## APS Workshop 3
## Developing Synchrotron Sample Environments to Study Next-generation Field-driven Device Physics

Location: Bldg. 401, Room A1100

Organizers: Philip Ryan (APS), Markys Cain (National Physical Laboratory, UK), and Paul Thompson (XMaS, European Synchrotron, France)

Condensed matter physics dominates our technological and economic trajectory, and new exotic phenomena are always considered in the light of applications. Strongly correlated electron physics is a particularly exciting subfield that promises many device variations. It relates to a large class of materials that show extraordinary coupling between several degrees of freedom, including electronic, structure, and magnetic orders. In fact, we are witnessing the emergence of novel device scenarios such as piezotransistors [1], magnetoelectronics [2,3], and mottronics [4], among others.

Microscopically, charge ties many of these behaviors together through piezoelectricity, magneto-electricity, magneto-caloric effects, pyro-electric effects, or multiferroicity. Macroscopically, strain through varying strictive forces acting over longer length scales linearly couples these order parameters, generating exciting possibilities for the innovative device engineer. Use of state-of-the-art synchrotron capabilities has already generated a deep understanding of these coupling properties, and as we develop ways to control these behaviors (either directly through applied fields or physically, i.e., through piezo-strain) we inadvertently generate a new paradigm of physical parameters to be explored. In addition, as we prepare the next generation of smaller and more coherent x-ray synchrotron sources, we are compelled to consider how to take advantage of these properties in order to probe and ultimately control such intricate and powerful phenomena.

It is the goal of this workshop to bring together experts with a strong emphasis on exciting topics regarding emerging device physics and additionally examples of more fundamental topics of strongly correlated phenomena. The resulting discussions will help to guide the APS to envision cutting-edge sample environments and supplemental *in situ* (x-ray) measurements that will augment experimental data and enrich our understanding of the underlying physics.

References

[1] National Physical Laboratory, "New global research effort to measure nanoscale strain," November 6, 2013, http://www.npl.co.uk/news/new-global-research-effort-to-measure-nanoscal....

[2] The Piezo Institute, "National Physical Laboratory: Metrology for Advanced Sensors through Engineered Magnetoelectric Coupling," http://www.piezoinstitute.com/about/current-research/metrology-for-advan....

[3] J.T. Heron et al., "Deterministic switching of ferromagnetism at room temperature using an electric field," *Nature* **516**, 370–373 (2014), doi:10.1038/nature14004.

[4] H. Yamada et al., "Ferroelectric Control of a Mott Insulator," *Scientific Reports* **3**, 2834 (2013), doi:10.1038/srep02834.

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**Tuesday, May 12 (all day)**

8:30 – 9:00 Philip Ryan (Argonne National Laboratory)
*Introductory Remarks*

| | |
|---|---|
| 9:00 – 9:40 | Markys Cain (National Physical Laboratory, UK)<br>*"Nanostrain Project" — Novel Electronic Devices Based on Control of Strain at the Nanoscale* |
| 9:40 – 10:00 | Glenn Martyna (IBM T.J. Watson Research Center)<br>*The Piezoelectronic Transistor: A Stress-driven Next-generation Transduction Device* |
| 10:00 – 10:35 | Break |
| 10:35 – 11:15 | Paul Evans (University of Wisconsin)<br>*Nanosecond Electric-field-driven Structural Phase Transitions in Complex Oxides* |
| 11:15 – 11:55 | Jacob Jones (North Carolina State University)<br>*New Direct Measurements of Polarizability Mechanisms in Ferroelectrics via Diffraction and Scattering* |
| 11:55 – 1:30 | Lunch |
| 1:30 – 2:10 | Siddharth Saxena (University of Cambridge, UK)<br>*Searching for Multivariable Quantum Criticality* |
| 2:10 – 2:50 | Peter Fischer (Center for X-ray Optics, Lawrence Berkeley National Laboratory)<br>*Magnetic Soft X-ray Spectro-microscopy: Seeing Nanoscale Magnetism in Action* |
| 2:50 – 3:20 | Break |
| 3:20 – 4:00 | Xavi Marti (Institute of Physics, Czech Republic)<br>*Antiferromagnetism: Applications of Invisible Magnets* |
| 4:00 | Sae Hwan Chun (Materials Science Division, Argonne National Laboratory)<br>*Static and Dynamic Magnetoelectric Effects in Multiferroic Hexaferrites* |

**Wednesday, May 13 half-day**

| | |
|---|---|
| 9:00 – 9:40 | Martin Holt (Center for Nanoscale Materials, Argonne National Laboratory)<br>*Strain Imaging of Nanoscale Semiconductor Heterostructures with X-ray Bragg Projection Ptychography* |
| 9:40 – 10:20 | Jian Liu (University of California, Berkeley)<br>*Toward* in situ *Control and Probe over Novel Magnetoelectric Effects in Magnetic Thin Films and Heterostructures* |
| 10:20 – 11:00 | Raegan Johnson (Sandia National Laboratory)<br>In situ *Electric Field Measurements of Ferroelectric Domain Wall Motion in PZT Thin Films* |
| 11:00 – 11:40 | Sean McMitchell (University of Liverpool, UK)<br>*Developing Traceable Links between Mesoscopic Strain and Crystallography through* in situ *Interferometrys* |
| 11:40 – 12:20 | Yong Choi (X-ray Science Division, Argonne National Laboratory)<br>*Interfacial Orbital Modification Probed by Polarization-dependent Anomalous X-ray Reflectivity* |
| 12:20 | Philip Ryan (Argonne National Laboratory)<br>*Wrap-up* |

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## Introductory Remarks

**Philip Ryan**

**Advanced Photon Source, X-ray Science Division, Argonne National Laboratory, Argonne, IL 60439**

I will present a brief vision of the future experimental environment given the anticipated coherence and spatial dimensions that will come with the APS-U project. The general condensed-matter end-user community may question how this affects their user relationship with the facility. My aim in these introductory remarks is to explore how the facility may assuage concerns of negative impacts and more importantly how we, the beamline scientist and user community, can take advantage of the improvements to the beam characteristics. In fact the net potential benefit to the broader community is quite exciting; however, we need to coordinate our efforts to maximize the optimal utilization of the future facility.

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## "Nanostrain Project" — Novel Electronic Devices Based on Control of Strain at the Nanoscale

**Markys Cain**

**National Physical Laboratory, Teddington, Middlesex, TW11 0LW, UK**

Faster, smaller and more energy efficient computing, based on miniature electronic devices, will benefit almost every industrial sector. Recently, materials such as piezoelectrics have been used to develop miniature electronics by allowing the control of properties at the nanoscale via the application of mechanical strain. Piezoelectric materials are uniquely capable of generating precisely defined strains down to very small length scales and are the technology driver for new types of electronic devices.

Currently, there is no measurement framework or facility for traceable measurement of the electromechanical coupling (shown as strain through application of voltage) in piezoelectric materials down to a size of 1 nm.

This project will develop traceable measurements of strain at lengths down to 1 nm and at high electric fields. These measurements need to be non-destructive and should be able to operate on the commercial scale. The results will help develop computing products based on the principles of functional materials such as piezoelectrics.

In this presentation I will explain the following key objectives of the scientific research project, with an emphasis on the first technical goal:

- Develop links between traceable mesoscale strain metrology and crystallographic strain via *in situ* interferometry and synchrotron x-ray diffraction. This provides for an assessment of the intrinsic piezo response to its, industrially highly relevant, extrinsic (domain mediated) response.
- Develop ultra-high spatial resolution (100 nm or less) optical methods of strain measurements using IR-SNOM by utilising the PTB synchrotron radiation facility (MLS) in Berlin as an IR light source.
- Develop traceable validation of macroscale strain metrology in destructive methods including Transmission Electron Microscopy (TEM) and novel holographic TEM, to map intra-grain residual and active (electric field induced) strains. The uncertainty caused by the additional strain from the preparation of TEM slices will also be investigated.
- Develop the multiphysics materials modelling to underpin all the experimental activities described above, considering both residual, process-related strains in thin film and nano/micro-scale released structures, and electrically driven strains in active devices.

WK3

## The Piezoelectronic Transistor: A Stress-driven Next-generation Transduction Device

**G.J. Martyna[1], P.M. Solomon[1], M. Copel[1], J. Chang[1], T.M. Shaw[1], R. Keetch[2], S. Trolier McKinstry[2], and D.M. Newns[1]**

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[2] Department of Materials Science and Engineering, Pennsylvania State University, University Park, PA 16802

We have invented a transduction based post-CMOS device based on a piezoelectrically driven metal insulator transition [1]. An input voltage pulse activates a piezoelectric element (PE) which transduces input voltage into an electro-acoustic pulse that in turn drives an insulator to metal transition (IMT) in a piezoresistive element (PR); the transition effectively transduces the electro-acoustic pulse to voltage. Using the known properties of *bulk* materials, we predict using modeling that the PET achieves multi-GHz clock speeds with voltages as low as 0.1 V and a large On/Off switching ratio ($\approx 10^4$) for digital logic [1]. The PET switch is compatible with CMOS-style logic. At larger scale the PET is predicted to function effectively as a large-area low voltage device for use in sensor applications.

PET device performance is enabled by the properties of two materials, a relaxor piezoelectric for the PE and a rare earth chalcogenide piezoresistor for the PR — provided the materials exhibit bulk properties at the nanoscale. Thus it is critical to investigate materials scaling using a combined theoretical/experimental approach. The development of thin film piezoresistive and piezoelectric materials and patterned structures, and associated characterization tools is presented, along with the theoretical models that yield insight into their behavior [2–4]. Integration of these novel materials into 3 evolutionary generations of PET devices, and device characterization, is given [5] to show that a proof of concept has been achieved.

*DARPA Mesodynamic Architectures Program under contract number N66001–11-C-4109.*

[1] "High Response Piezoelectric and Piezoresistive Materials for Fast, Low Voltage Switching: Simulation and Theory of Transduction Physics at the Nanometer-Scale," D.M. Newns, B.G. Elmegreen, X.-H. Liu, and G.J. Martyna, *Adv. Mat.* **24**, 3672 (2012).

[2] "Giant Piezoresistive On/Off Ratios in Rare-Earth Chalcogenide Thin Films Enabling Nanomechanical Switching," M. Copel, G.J. Martyna, and D.M. Newns et al., *Nano Lett.* **13**, 4650 (2013).

[3] "Anisotropic strain in SmSe and SmTe: implications for electronic transport," M.A. Kuroda, Z. Jiang, M. Povolotskyi, G. Klimeck, D.M. Newns, and G.J. Martyna, *Phys. Rev. B.* **90**, 245124 (2014).

[4] "Lateral scaling of PMN-PT thin films for piezoelectric logic," R. Keech, S. Shetty, M.A. Kuroda, X.-H. Liu, G.J. Martyna, D.M. Newns, and S. Trolier-McKinstry, *J. Appl. Phys.* **115**, 234106 (2014).

[5] "Pathway to the PiezoElectronic Transduction Logic Device," accepted *NanoLetters* (2014).

WK3

## Nanosecond Electric-field-driven Structural Phase Transitions in Complex Oxides

**Paul G. Evans**

Materials Science and Engineering, University of Wisconsin-Madison, Madison, WI 53706

Complex oxide electronic materials, including ferroelectric and multiferroics, can exhibit large responses to externally applied fields when the system has a composition or external stresses that place the system near a boundary between structural phases. The development of time-resolved x-ray diffraction and scattering techniques allows us to probe the dynamics and energetics of piezoelectric distortion and phase transformation in complex oxide electronic materials at such boundaries, with both nanosecond time resolution and a high level of structural precision. Compressive epitaxial stresses can place thin films of the multiferroic oxide $BiFeO_3$ at a boundary between phases with different structural extent. The electric-field driven transition exhibits contributions from both stably switching and reversible components, and can occur at times as short as tens of nanoseconds. Comparison with density functional theory results shows that the transition can be understood by comparing the free energies of the piezoelectrically distorted structures. Control of this transition, and other field-driven phase transition phenomena, has the potential to provide an additional route to the control of electronic and magnetic properties of complex oxides.

WK3

## New Direct Measurements of Polarizability Mechanisms in Ferroelectrics via Diffraction and Scattering

**J.L. Jones[1], T.-M. Usher[1], C.C. Chung,[1] and C.M. Fancher[1], I. Levin[2], S. Brewer,[3] and N. Bassiri-Gharb[3]**

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[3]G.W. Woodruff School of Mechanical Engineering, Georgia Institute of Technology, Atlanta, GA 30332

The functionality of many dielectric and ferroelectric materials is dependent on the external application of electric fields. Characterizing the response of the material at length scales ranging from sub-nanometer to micrometer is important for understanding and engineering these functionalities. For field-induced strain, for example, both the contribution of the intrinsic piezoelectric effect (originating at sub-nanometer dimensions) and ferroelectric/ferroelastic domain wall motion (at the nanometer length scale) contribute to strain. For both polarizability and permittivity, it is important to characterize and understand the field-induced response of the local structure (i.e., ionic polarizability) and changes in the domain structure (i.e., dipolar). In this talk, we present the new development of two different methods to characterize both local structure and domain structure changes during field application and the contribution of these mechanisms to polarization.

In the first method, we have used x-ray diffraction *in situ* during application of electric fields in $BaTiO_3$ to determine the contribution of ferroelectric/ferroelastic (i.e., non-180°) domain walls to the polarization. We have used a complementary analysis approach to further determine the contribution from ferroelectric (i.e., 180°) domain walls to the polarization.

In the second method, we have used *in situ* total x-ray scattering during application of electric fields and determined pair distribution functions (PDFs). The field-dependent PDFs enable the assessment of local structure changes in $BaTiO_3$ and $Na_{0.5}Bi_{0.5}TiO_3$. A unique dipolar mechanism is observed at the unit-cell level in $Na_{0.5}Bi_{0.5}TiO_3$ which involves rearrangements of $Bi^{3+}$ displacements. In contrast, $BaTiO_3$ exhibits a smaller local scale response to the electric field which is nonetheless consistent with piezoelectric strain.

The results are interpreted holistically to enable ascription of dominant mechanisms of polarizability in dielectric and ferroelectric materials. For example, the first and second largest contributions to polarizability in $BaTiO_3$ are 180° domain wall motion and non-180° domain wall motion. The new characterization methods can be readily applied to other materials.

WK3

## Searching for Multivariable Quantum Criticality

**Siddharth S. Saxena**

Cavendish Laboratory, Madingley Road, Cambridge, CB3 0HE, Great Britain

This talk will discuss pressure induced phenomena in the vicinity of magnetic quantum phase transitions and Quantum Criticality in Metallic and Insulating Systems.

Materials tuned to the neighbourhood of a zero temperature phase transition often show the emergence of novel quantum phenomena. Much of the effort to study these new emergent effects, like the breakdown of the conventional Fermi-liquid theory in metals has been focused in narrow band electronic systems. Ferroelectric crystals provide a very different type of quantum criticality that arises purely from the crystalline lattice. In many cases the ferroelectric phase can be tuned to absolute zero using hydrostatic pressure. Close to such a zero temperature phase transition, the dielectric constant and other quantities change into radically unconventional forms due to the fluctuations experienced in this region. The simplest ferroelectrics may form a text-book paradigm of quantum criticality in the solid-state where there are no complicating effects of electron damping of the quantum charge fluctuations. We present low temperature high precision data demonstrating these effects in pure single crystals of $SrTiO_3$ and $KTaO_3$. We outline a model for describing the physics of ferroelectrics close to quantum criticality and highlight the expected 1/T2 dependence of the dielectric constant measured over a wide temperature range at low temperatures. In the neighbourhood of the quantum critical point we report the emergence of a small frequency independent peak in the dielectric constant at approximately 2K in $SrTiO_3$ and 3K in $KTaO_3$. Looking to the future, we imagine that quantum paraelectric fluctuations may lead to new low temperature states and mediate novel interactions in multi-ferroic systems (e.g., $EuTiO_3$) and ferroelectric crystals supporting itinerant electrons.

WK3

## Magnetic Soft X-ray Spectro-microscopy: Seeing Nanoscale Magnetism in Action

**Peter Fischer**

**Center for X-ray Optics, Lawrence Berkeley National Laboratory, Berkeley, CA 94720**
**Physics Department, University of California, Santa Cruz, CA 94056**

The era of nanomagnetism, which aims to understanding and controlling magnetic properties and behavior on the nanoscale, is currently expanding into the mesoscale [1], which will harness complexity and novel functionalities, which are essential parameters to meet future challenges in terms of speed, size and energy efficiency of spin driven devices. Multimodal characterization techniques, such as tomographic magnetic imaging and investigations of spin dynamics down to fundamental magnetic length and time scales with elemental sensitivity in emerging multi-component materials will enable future scientific breakthroughs. I will review recent developments with full-field magnetic soft x-ray transmission microscopy [2] to study spin configurations in magnetic nanotubes [3], the stochastic behavior in vortices [4] and the local distribution of magnetic properties near domain walls [5].

*This work was supported by the Director, Office of Science, Office of Basic Energy Sciences, Materials Sciences and Engineering Division, of the U.S. Department of Energy under Contract No. DE-AC02-05-CH1123 and by the Leading Foreign Research Institute Recruitment Program (Grant No. 2012K1A4A3053565) through the National Research Foundation of Korea (NRF) funded by the Ministry of Education, Science and Technology (MEST).*

[1] R. Service, *Science* **335** 1167 (2012).

[2] P. Fischer, *Materials Science & Engineeering* **R72** 81–95 (2011).

[3] M.-Y. Im et al., *Nature Communications* **5** 5620 (2014).

[4] R. Streubel, *Adv. Mater.* **26** 316 (2014).

[5] M.J. Robertson et al., *J Appl. Phys.* **117** 17D145 (2015).

WK3

## Antiferromagnetism: Applications of Invisible Magnets

**X. Marti[1,2], I. Fina[3,4], and T. Jungwirth[1,5]**

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[5] School of Physics and Astronomy, University of Nottingham, Nottingham NG7 2RD, UK

In 1970, at the time when compact cassettes made it to the market after a century of experimenting with ferromagnetic storage, the Nobel Prize was awarded for the "*fundamental work and discoveries concerning antiferromagnetism.*" Louis Neel pointed out in his Nobel lecture that while abundant and interesting from a theoretical viewpoint, antiferromagnets did not seem to have any applications. Indeed, the alternating directions of magnetic moments on individual atoms and the resulting zero net magnetization make antiferromagnets hard to control by tools common in ferromagnets. While preoccupied with the inherent difficulties to read and write magnetic information in antiferromagnets, scientists and engineers have largely overlooked the positive sides of antiferromagnets as being magnetically invisible [1] and uniquely robust against magnetic perturbations [2]. In this talk, we will review recent developments in the emerging field of antiferromagnetic spintronics, retracing the footsteps of the ferromagnetic based technologies. We will examine the path starting from seminal basic science experiments that took place at the Advanced Photon Source down to a survey of the potential markets for the coming up *invisible magnetism*.

[1] https://www.youtube.com/watch?v=X1Ft_OnRaq4.

[2] https://www.youtube.com/watch?v=HWZLJ02sb0U.

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## Static and Dynamic Magnetoelectric Effects in Multiferroic Hexaferrites

**Sae Hwan Chun**

**Materials Science Division, Argonne National Laboratory, Argonne, IL 60439**

Multiferroics, wherein magnetism and ferroelectricity coexist, are of great interest for the prospect of new multifunctional devices by using magnetoelectric (ME) effects through the cross-coupling between the magnetic and electric properties. In most multiferroics currently known, however, controlling electric polarization with magnetic field or magnetization with electric field has been realized only at low temperatures. In addition, their ME susceptibilities are too small for practical applications. Hence, it is essential to improve both the operating temperature and the ME sensitivity of magnetic ferroelectrics for use in ME devices. Investigating the multiferroic hexaferrites, we discovered a novel chemical route effectively tailoring the electric polarization induced by a low magnetic field in $(Ba,Sr)_2Zn_2Fe_{12}O_{22}$ ($Zn_2Y$-type) hexaferrite by Al-substitution to possess a giant magnetoelectric susceptibility (MES) [1]. Furthermore, in $(Ba,Sr)_3Co_2Fe_{24}O_{41}$ ($Co_2Z$-type) hexaferrite single crystals with large MES, we realized the control of magnetization by an electric field at room temperature [2]. In addition to those static ME properties, a dynamic ME effect, electric-dipole-active magnon resonance in THz frequency range, is found in the $Co_2Z$-type hexaferrite, exhibiting the spectral weight even at room temperature [3]. The unprecedented supreme static and dynamic ME phenomena in the hexaferrites may provide a pathway to overcome the challenge in application of multiferroics for real devices.

[1] S.H. Chun et al., *Phys. Rev. Lett.* **104**, 037204 (2010).

[2] S.H. Chun et al., *Phys. Rev. Lett.* **108**, 177201 (2012).

[3] S.H. Chun et al., in preparation.

WK3

## Strain Imaging of Nanoscale Semiconductor Heterostructures with X-ray Bragg Projection Ptychography

**M.V. Holt[1], S.O. Hruszkewycz[2], C.E. Murray[3], J.R. Holt[4], D.M. Paskiewicz[2], and P.H. Fuoss[2]**

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We report the imaging of nanoscale strain distributions in complementary components of lithographically engineered epitaxial thin film semiconductor channel heterostructures using synchrotron x-ray Bragg Projection Ptychography (BPP). A new phase analysis technique applied to the reconstructed BPP phase images from two laterally adjacent, stressed materials produced lattice strain and lattice rotation maps with a spatial resolution of ~15 nm, a strain sensitivity of better than 0.01%, and an angular resolution of ~0.1 mrad [1].

Bragg projection ptychography is a coherent diffraction x-ray imaging technique capable of mapping structural perturbation, such as strain, in single crystal thin films with nanoscale spatial resolution [2,3]. In this study, analysis of the orthogonal derivatives of the reconstructed phase maps provides insight into two distinct lattice responses that quantitatively agree with linear elastic predictions. This demonstrates that Bragg ptychography can be used to quantitatively visualize extremely subtle lattice perturbations at the nanoscale under realistic conditions without sectioning or otherwise modifying the boundary conditions of the sample.

[1] M.V. Holt et al., *Phys. Rev. Lett.* **112**, 165502 (2014).
[2] S.O. Hruszkewycz et al., *Nano Lett.* **12**, 5148 (2012).
[3] S.O. Hruszkewycz et al., *Phys. Rev. Lett.* **110**, 177601 (2013).

WK3

## Toward *in situ* Control and Probe over Novel Magnetoelectric Effects in Magnetic Thin Films and Heterostructures

**Jian Liu**

Department of Physics, University of California, Berkeley, CA 94720
Materials Science Division, Lawrence Berkeley National Laboratory, Berkeley, CA 94720

Magnetoelectric effects, which couple the spin and charge degree of freedom, are the key to the great success of various magnetic material-based technologies, such as recording and memories. The development of the next generation of magnetic devices with improved performance or new functionalities relies on creating, understanding, and controlling novel magnetoelectric effect/coupling. As a class of materials where the magnetic and electronic properties are tightly related, complex oxides present great potential in this area. The challenge, however, has been imposed by the fact that most magnetic oxides are antiferromagnetic and difficult to harness or probe at nanoscale. In this talk, I will show some of our recent work on different magnetoelectric effects in oxide heterostructures with antiferromagnetism as a key component. In particular, results that utilize synchrotron-based probes will be discussed.

WK3

## *In situ* Electric Field Measurements of Ferroelastic Domain Wall Motion in PZT Thin Films

**Raegan L. Johnson-Wilke [1], Margeaux Wallace[2], Rudeger H.T. Wilke [1], Giovanni Esteves[3], Jacob Jones[3], and Susan Trolier-McKinstry[2]**

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Lead zirconate titanate (PZT) thin films are used in microelectromechanical systems (MEMS) due to their large piezoelectric response. In order to quantitatively study this response, *in situ* electric field measurements were performed at the Advanced Photon Source at Argonne National Laboratory. The electromechanical response in PZT is a result of both the intrinsic (lattice) piezoelectric effect as well as the motion of ferroelectric and ferroelastic domain walls (extrinsic effect). In-depth studies of the intrinsic and extrinsic effects in bulk materials has been performed on a number of ceramic materials, however, thin films present a challenge due to the significantly smaller volume of material. This talk will outline the challenges and describe the experimental setup used to perform the *in situ* measurements on PZT thin films. In addition, the work presented will describe direct measurements of ferroelastic domain wall motion in 2-μm thick {001} oriented PZT (30/70) films under different release states. Films that were fully clamped to the underlying substrate are compared with films that are up to 75% released from the wafer. The released films showed significantly larger domain wall motion response based on the changes in peak intensity between the 200 and 002 diffraction peaks. Quantitative details of the influence of the release state and electric field will be discussed.

WK3

## Developing Traceable Links between Mesoscopic Strain and Crystallography through *in situ* Interferometry

**S.R.C. McMitchell[1, 2], P. Thompson[1, 2], C. Lucas[1, 2], C. Vecchini[3], J. Wooldridge[3], M. Stewart[3], A. Muniz-Piniella[3], M. Cain[3], and T. Hase[1, 4]**

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Recently, there has been considerable research effort on understanding the complex interplay between material structure and the internal strain in piezo and ferroelectrics, and multiferroics. This is a key factor in the functional efficiency of devices.

Of particular relevance is the correlation between strain and electric polarisation, which is being exploiting to develop a novel Piezoelectric-Effect-Transistor (PET), which offers a possible route to replace current CMOS technology. To aid the development of this transformative technology, several European national laboratories, academic and commercial partners formed a consortium funded through the European Metrology Research Programme (EMRP) Project IND54 Nanostrain. The EMRP is jointly funded by the EMRP participating countries within EURAMET and the European Union.

The operation of the PET will be controlled through application of an electric field. It is therefore imperative to investigate the physical deformation and strain state that occurs under applied electric fields *in situ* and *in operando.* We have incorporated a dual-beam optical interferometer onto the XMaS beamline at the ESRF. *In situ* polarisation, lattice parameter, and deformation measurements allow new insights into the correlation between induced strain and material properties in piezoelectrics. We will detail experimental strategies employed to reduce noise and show results from both static (d.c.) and dynamic cycles (up-to 55Hz) of electric field. Standard piezoelectric single crystals and thin films were used to develop quantitative and traceable metrologies for the precise determination of bulk and atomic strain within these structures. The addition of magnetic field and temperature dependence is also discussed.

WK3

## Interfacial Orbital Modification Probed by Polarization Dependent Anomalous X-ray Reflectivity

**Yong Choi**

Advanced Photon Source, X-ray Science Division, Argonne National Laboratory, Argonne, IL 60439

In a lattice matched multilayer structure, materials with similar structures but dissimilar properties can be forced to coexist at the interfaces. Numerous recent studies have shown novel interfacial properties that are not intrinsic to either of the constituent materials. We investigate the role of interfacial modification in the metal-to-insulator transition observed from superlattices of two $RNiO_3$ (R=rare earth) with different transition behaviors, using a combined approach between spectroscopy and scattering to take advantage of the element-specificity of x-ray absorption near edge spectroscopy and the enhanced interfacial sensitivity of x-ray reflectivity.

# Tuesday, May 12
# Facility-specific Workshops

## CNM Workshop 4
## Exploring the Flatland of 2D Materials for Tribological Manipulation

Location: Bldg. 402, Room E1100/E1200

Organizers: Anirudha Sumant (CNM), Diana Berman (CNM), and Ali Erdemir (Argonne National Laboratory)

The purpose of this workshop is to bring together experts from academia, national labs, and industry to discuss the latest developments in the synthesis, functionalization, and characterization of nanostructured 2D materials, with an emphasis on uncovering their unique tribological properties at the micro/nano-scale and to develop the next generation of energy-efficient moving systems based on the unique wear/friction performance of these 2D materials.

The materials of interest include graphene, boron nitride, molybdenum disulfide, maxenes, and others. With recent fast-paced advances in the development of 2D materials and understanding of their mechanical and tribological properties, we now have a completely new perspective on how they behave as a lubricant compared to traditional thin film or bulk solid lubricants.

The exceptional physical, chemical, mechanical, electrical, and tribological properties of 2D materials, combined with the ability to apply or introduce them on the contacting interfaces of tribological systems, make them ideal new solid lubricant materials for a wide variety of applications, ranging from nano/micro scales (NEMS/MEMS) to macro/meso scales (e.g., moving electrical contacts, sliding/rolling contacts, rotating contacts, bearings, etc.).

The Center for Nanoscale Materials (CNM) at Argonne has expertise and state-of-the-art facilities for the synthesis, functionalization, and characterization of 2D materials. In the last few years the CNM has developed a strong research program in fundamental studies of the physical properties of these materials, with particular emphasis on understanding their tribological properties at the nano, micro, and macro scale.

This workshop will include topics covering fundamental studies on the development of 2D materials and their systematic characterization to understand their structural, surface chemical, mechanical, and tribological properties. It will also cover theoretical and modeling approaches that can provide in-depth understanding of tribo-physical and chemical interactions with the substrate and counterface materials under severe contact pressure and shear stress, issues related to dispersibility, substrate interactions, and possibilities for industrial applications.l

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|---|---|
| 8:30 – 8:45 | Anirudha Sumant (Argonne National Laboratory)<br>*Welcome & Introductory Remarks* |
| 8:45 – 9:25 | Robert Carpick (University of Pennsylvania)<br>*Mechanisms of Atomic-scale Friction for 2D Materials* |
| 9:25 – 10:05 | James Batteas (Texas A&M University)<br>*2D or not 2D? The Impact of Nanoscale Roughness and Substrate Interactions on the Tribological Properties of Graphene* |
| 10:05 – 10:35 | Break |
| 10:35 – 11:15 | Elena Polyakova (Graphene Supermarket Inc.)<br>*The Use of Graphene and Other 2D Materials in 3D Printing* |
| 11:15 – 12:00 | Alex Smolyanitsky (National Institute of Standards and Technology)<br>*The Effects of Intrinsic Rippling on the Frictional Properties of Atomically Thin Membranes: A Pathway to Real-time Controllable Surface Properties* |
| 12:00 – 1:30 | Lunch |
| 1:30 – 2:10 | Yury Gogotsi (Drexel University)<br>*MXenes and MXene-polymer Composites: Manufacturing, Nanoindentation and Nanotribology* |
| 2:10 – 2:50 | Christopher Muratore (University of Dayton)<br>*Ultra-thin Transition Metal Dichalcogenides Grown by Physical Vapor Deposition* |
| 2:50 – 3:20 | Break |
| 3:20 – 4:00 | Nikhil Koratkar (Rensselaer Polytechnic Institute)<br>*Wear in Graphene-polymer Composites* |
| 4:00 – 4:40 | Diana Berman (Argonne National Laboratory)<br>*Tapping into Graphene's Potential as a Solid Lubricant* |
| 4:40 | Ali Erdemir (Argonne National Laboratory)<br>*Wrap-up and concluding remarks* |

WK4

## Mechanisms of Atomic-scale Friction for 2D Materials

**Robert W. Carpick**

University of Pennsylvania, Philadelphia, PA 19104

Two-dimensional materials provide a rich playground for exploring new and unexpected physical phenomena, including tribological. This talk will focus on interfacial friction and adhesion behavior of nanoscale contacts with such materials. We find that single layer graphene reduces friction by an order of magnitude for surfaces including copper [1]. However, the friction reduction is layer dependent: for contacts to graphene, $MoS_2$, $NbSe_2$, and BN, the friction force exhibits a significant dependence on the number of 2-D layers [2] which we attribute to an out-of-plane "puckering" deformation that occurs when the 2-D material is weakly bound to its substrate. However, adhesive behavior does not follow this dependence. Instead, we find that sliding can induce an increased adhesive

force due to local delamination of the topmost layer of graphene [3]. Finally, we observe a large, order-of-magnitude increase in friction that occurs when graphene is fluorinated [4]. This result is interpreted in the context of the Prandtl-Tomlinson model of stick-slip friction, where static friction arises from the high electronic roughness of fluorinated graphene.

[1] Frictional behavior of atomically thin sheets: Hexagonal-shaped graphene islands grown on copper by chemical vapor deposition. P. Egberts, G.H. Han, X.Z. Liu, A.T. Johnson, and R.W. Carpick. *ACS Nano* **8**, 5010–21 (2014). (http://dx.doi.org/10.1021/nn501085g.)

[2] Frictional characteristics of atomically thin sheets. C. Lee, Q. Li, W. Kalb, X.-Z. Liu, H. Berger, R.W. Carpick, and J. Hone. *Science* **328**, 76–80 (2010). (http://dx.doi.org/10.1126/science.1184167.)

[3] Nanoscale adhesive properties of graphene: The effect of sliding history. X.-Z. Liu, Q. Li, P. Egberts, and R.W. Carpick. *Adv. Mat. Interf.* **1**, 1300053 (2014). (http://dx.doi.org/10.1002/admi.201300053.)

[4] Fluorination of graphene enhances friction due to increased corrugation. Li, Q., Liu, X.Z., Kim, S.P., Shenoy, V.B., Sheehan, P.E., Robinson, J.T., and Carpick, R.W. *Nano Letters* **14**, 5212–5217 (2014). (http://dx.doi.org/10.1021/nl502147t.)

WK4

## 2D or not 2D? The Impact of Nanoscale Roughness and Substrate Interactions on the Tribological Properties of Graphene

**Jessica Spear and James Batteas**

**Departments of Chemistry and Materials Science and Engineering, Texas A&M University, College Station, TX 77843**

Control of friction and wear is a ubiquitous challenge in numerous machined interfaces ranging from biomedical implants, to engines, to nano- and micro-scaled electromechanical systems (MEMS) devices. A key challenge in developing boundary lubrication schemes for such systems is how to reduce wear at the rough surfaces of such devices, where nanoscaled asperities dominate the interfacial contacts. The robust mechanical properties of two-dimensional (2D) nanomaterials, such as graphene, has made it a material of interest for modifying surface frictional properties. While graphene can readily adapt to surface structure on the atomic scale, when deposited on substrates with nanoscopic roughness (~ 10 — 20 nm rms as is common in many machined interfaces) a conformal coating cannot be fully formed due to competition between adhesion to substrate nanoscopic asperities and the bending strain of the graphene. This often leaves a mixture of supported and unsupported regions which respond differently to applied load. Here we describe a combination of AFM nanomechanical and confocal Raman microspectroscopy studies of graphene on silica surfaces with controlled nanoscopic roughness to examine the how this impacts the frictional properties of graphene. Composite interfaces where graphene is supported on self-assembled alkylsilane monolayers will also be described along with the synergistic influence of such mixed interfaces on the frictional properties of the surface.

WK4

## The Use of Graphene and Other 2D Materials in 3D Printing

**Elena Polyakova**

**Graphene 3D Lab, Calverton, NY 11933**

The 3D printing market is expected to grow quickly over the course of the next few years, without factoring in the likelihood of advances in both printer and filament technologies. One advance is the use of graphene, a 2D material known for its extraordinary attributes, in filament production. Adding graphene to filaments increases the strength and adds conductivity to end-products made via 3D printing — a major advancement when considering using 3D printing for energy storage, electronic, and other commercial applications. I will overview the recent progress made in this area at Graphene 3D Lab, as well as the applications of other 2D materials for 3D printing.

WK4

## The Effects of Intrinsic Rippling on the Frictional Properties of Atomically Thin Membranes: A Pathway to Real-time Controllable Surface Properties

**Alex Smolyanitsky**

**National Institute of Standards and Technology, Boulder, CO 80305**

Frictional properties of atomically thin layers and lamellar materials have recently become a topic of significant research interest. As a result, graphene and its chemical derivatives have been suggested as revolutionary solid-state lubricants with unique tribological properties. Possible uses range from novel methods of determining exfoliation energies to nanoelectromechanical (NEMS) applications. Most studies to date have focused on the structural properties, including the effects of chemical modification and substrate interactions.

The first simulated friction force microscopy study of the dependence of the frictional properties of free-standing graphene as a function of temperature is presented. In contrast with the long-standing theory for dry surfaces of solids, sliding friction is predicted to *both increase and decrease* with increasing temperature. The qualitative behavior is demonstrated to depend on the local lateral strains, normal contact force, sample size, and the lateral scanning rate. Our observations are likely due to the intrinsic presence of dynamic flexural ripples in atomically thin layers at finite temperatures. The effect of random thermally excited waves on dry friction suggests the concept of real-time controllable dynamic roughness in free-standing atomically thin layers via imposed lateral strains or externally excited low-energy flexural waves.

The effect of externally engineered rippling content on the frictional properties of free-standing atomically thin layers is described. It is shown that, depending on the excitation amplitude and frequency, sliding friction at identical normal loads can be modified significantly. In addition to possible uses in NEMS applications, the observed real-time control of the effective surface "roughness" may be employed for controllable gas and fluid passage at the nanoscale, as well as for separation of species in gas or fluid mixtures.

WK4

## MXenes and MXene-polymer Composites: Manufacturing, Nanoindentation and Nanotribology

**Babak Anasori[1], Xin Liu[2], Michel Barsoum[1], Robert W. Carpick[2], and Yury Gogotsi[1]**

[1]Department of Materials Science and Engineering, and A.J. Drexel Nanomaterials Institute, Drexel University, Philadelphia, PA 19104

[2]Department of Mechanical Engineering and Applied Mechanics, University of Pennsylvania, Philadelphia, PA 19104

Two-dimensional (2D) solids – the thinnest materials available to us – offer unique properties and a potential path to device miniaturization. The most famous example is graphene, which is an atomically thin layer of carbon atoms bonded together in-plane with $sp^2$ bonds. Recently, an entirely new family of 2D solids – transition metal carbides ($Ti_2C$, $Ti_3C_2$, $Nb_4C_3$, etc.) and carbonitrides – was discovered by our group [1,2]. Selective etching of the A-group element from a MAX phase results in formation of 2D $M_{n+1}X_n$ solids, labeled "MXene." We have produced about 15 different carbides and carbonitrides [2–6]. Their elastic constants along the basal plane are expected to be similar to or even higher than that of the binary carbides. Oxygen or OH terminated MXenes, are hydrophilic, but electrically conductive.

Here we report on manufacturing of $Ti_3C_2$-PVA composite films [7] and initial studies of their mechanical properties using nanoindentation. At 22±1 GPa, the elastic modulus of the 60 wt.% $Ti_3C_2$-PVA composite film was measured to be significantly higher than its end members (pure MXene or pure PVA). In addition, tribological properties of different MXenes and effect of the surface termination on friction were studied using atomic force microscopy.

[1] M. Naguib et al., *Advanced Materials* **23** (37), 4207–4331 (2011).
[2] M. Naguib et al., *ACS Nano* **6** (2), 1322–1331 (2012).
[3] O. Mashtalir et al., *Nature Communication* **4**, 1716 (2013).
[4] M. R. Lukatskaya et al., *Science* **341**, 1502–1505 (2103).
[5] M. Naguib et al., *Advanced Materials* **26**, 992–1005 (2014).
[6] M. Ghidiu et al., *Nature* **516**, 78–81 (2014).
[7] Z. Ling et al., *PNAS* **111** (47) 16676–16681 (2014).

WK4

## Ultra-thin Transition Metal Dichalcogenides Grown by Physical Vapor Deposition

**C. Muratore[1] and A.A. Voevodin[2]**

[1]Department of Chemical and Materials Engineering, University of Dayton, Dayton, OH 45469
[2]Materials and Manufacturing Directorate, Air Force Research Laboratory, Wright-Patterson AFB, OH 45433

Physical vapor deposition (PVD) is a thin film growth technique allowing exquisite control of structure and composition and freedom from many thermodynamic constraints. This is evidenced by the common use of sputter deposition in a significant fraction of the total fabrication steps required to produce ubiquitous commercial electronic devices. By employing well-known theories of film growth and making the necessary conditions a reality through the use of surface engineering technology, thin film microstructures become very tailorable via PVD. This is of particular importance in the field of two dimensional (2D) materials, where processing of continuous and uniform films with thicknesses on the order of 1 nm is currently a major challenge. Transition metal dichalcogenides (TMDs), such as $MoS_2$ are currently under extensive study as high-performance 2D semiconductors. Using PVD techniques, which are easy to integrate into existing semiconductor device fabrication processes, 2D TMDs can be grown on diverse substrates including $SiO_2$, graphene, metals, and polymers. This is surprising as it is more common for such thin films to form isolated islands on substrates. Using a variety of *in situ* and conventional *ex situ* materials characterization tools such as Raman spectroscopy and x-ray photoelectron spectroscopy, the mechanisms governing continuous growth of TMDs have been examined for all of the substrates listed above. These substrates possess a broad range of surface energies. It appears that under some conditions a continuous metal monolayer is formed initially on higher surface energy substrates, and normal TMD growth continues on that interfacial metal layer. This is typically observed at high temperature where preferential desorption of the chalcogen atoms during growth is enhanced. How the crystalline domain size within 1–5 nm thick TMD films can be manipulated during PVD growth over at least one order of magnitude, and how the domain boundary density affects electronic, thermal, surface reactivity, and mechanical properties in diverse 2D devices.

WK4

## Wear in Graphene-polymer Composites

**Nikhil Koratkar**

Department of Mechanical, Aerospace and Nuclear Engineering and Department of Materials Science and Engineering, Rensselaer Polytechnic Institute, Rensselaer Polytechnic Institute, Troy, NY 12180

Polytetrafluoroethylene (PTFE) is one of the most widely used solid lubricants but suffers from a high wear rate which limits its applications. Here we report four orders of magnitude reduction in the steady state wear rate of PTFE due to graphene additives. The wear rate of unfilled PTFE was measured to be ~$0.4 \times 10^{-3}$ $mm^3$/Nm which is reduced to ~$10^{-7}$ $mm^3$/Nm by the incorporation of 10 weight % of graphene platelets. We also performed a head-to-head comparison of wear rate with graphene and micro-graphite fillers at the same weight fractions. In general, we find that graphene fillers gave 10 to 30 times lower wear rates than micro-graphite at the same loading fraction. Scanning electron microscopy analysis indicated noticeably smaller wear debris size in the case of graphene/PTFE composites indicating that graphene additives are highly effective in regulating debris formation in PTFE leading to reduced wear. We systematically varied the weight fraction and thickness of the graphene platelets and find that when the wear results are normalized by the filler specific surface area per unit mass, the results for the various graphene samples fall on a master curve. We use percolation theory to explain these results.

WK4

## Tapping into Graphene's Potential as a Solid Lubricant

**Diana Berman[1], Sanket A. Deshmukh[1], Subramanian K.R.S. Sankaranarayanan[1], Ali Erdemir[2], and Anirudha V. Sumant[1]**

[1] Nanoscience and Technology Division, Argonne National Laboratory, Argonne, IL 60439
[2] Energy Systems Division, Argonne National Laboratory, Argonne, IL 60439

In recent years minimizing friction and wear-related failures in mechanical/tribological and electronic subsystems remains as one of the greatest challenges in today's moving mechanical assemblies. It is estimated that nearly 1/3 of the fuel used in automobiles is spent to overcome friction. Accordingly, the search continues for novel materials, coatings, and lubricants (both liquid and solid) that can potentially reduce friction and wear.

In this study we show, that few layers of graphene are able to drastically reduce friction (by factors of 4–5) and wear (by as much as 4 orders of magnitude) of sliding metallic surfaces in both humid and dry environment. This tribological behavior of graphene is strikingly different from its bulk counterpart graphite. Additionally, we observed that the lifetime of such few-layer graphene coating improves significantly, when tested in hydrogen environment [1]. Separate studies on evaluating the electrical contact properties of these graphene sheets demonstrated that graphene, as a two-dimensional material, shears easily during mechanical sliding tests even under high contact pressures, while retaining excellent electrical conductivity of metal-metal contacts for thousands of sliding passes [2]. We elucidate the mechanism of wear/friction of graphene using reactive molecular dynamic simulation. Our findings demonstrate that tuning the atomistic scale chemical interactions holds the promise of realizing extraordinary tribological properties of monolayer graphene coatings.

*Use of the Center for Nanoscale Materials was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.*

[1] D. Berman, S. Deshmukh, S. Sankaranarayanan, A. Erdemir, and A.V. Sumant, "Extraordinary macroscale wear resistance of one atom thick graphene layer," *Advanced Functional Materials* **24**, 6640–6646 (2014).

[2] D. Berman, A. Erdemir, and A.V. Sumant, "Graphene as a protective coating and superior lubricant for electrical contacts," *Applied Physics Lett.* **105**, 231907 (2014).

# Wednesday, May 13
# Facility-specific Workshops

## APS Workshop 6
## APS Upgrade: Planning the First Experiments

Location: Bldg. 402, Lecture Hall

Organizers: Eric Landahl (DePaul University) and Stefan Vogt (Argonne National Laboratory)

The purpose of this workshop is to discuss the opportunity to create Partner User Proposals (PUPs) for developing experimental systems, methods, and technologies needed to lay the foundation for the first experiments at the APS Upgrade. It is expected that a significant fraction of beam time over the next several years will need to be dedicated to the efforts of the Upgrade Partner Users.

Following a brief status update on the Upgrade, including a discussion of anticipated performance parameters, this workshop will break into self-organized groups of interested participants. An APS beamline staff member will serve as moderator for each working group and will be able to provide additional beamline-related information. PUPs resulting from this workshop might include the following:

Development of new model systems or samples that will take advantage of the improved coherence of the upgraded storage ring. PUPs are encouraged that will exploit existing capabilities to conduct preliminary studies on new samples that are candidates for the first experiments at the Upgrade

Development of experimental or analysis techniques that will reach their full potential using the improved coherence of the upgraded storage ring. PUPs might include exploiting coherence in existing techniques (e.g., applying coherence methods to macromolecular crystallography or EXAFS) or in testing entirely new approaches to coherent x-ray science.

Development of technologies necessary for successful use of the upgraded storage ring. PUPs might be used to provide beam time for testing optics, detectors, or data acquisition aspects of anticipated first experiments.

PUPs contribute to the development of the facility in exchange for a set amount of beam time. Further information is available on the PUP web page.

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8:30 – 8:40 Eric Landahl
*Welcome & Introduction*

8:40 – 8:55 Stuart Henderson
*Overview of the APS Upgrade*

8:55 – 9:20 Stefan Vogt
*The APS Upgrade: Unique Opportunities*

9:20 – 9:30 Dennis Mills
*The Partner User Proposal Process*

9:30 – 10:15 Open forum and single-slide staff presentations to seed breakout discussions groups

10:15 – 10:45 Coffee Break

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| 10:45 – 12:00 | Breakout discussions groups (locations announced during workshop) |
| 12:00 | Adjourn |

# Wednesday, May 13
# Facility-specific Workshops

## APS/CNM Workshop 7
## Experimental and Computational Challenges of *in situ* Multimodal Imaging of Energy Materials

Location: Bldg. 401, Room A1100

Organizers: Yuzi Liu (CNM), Yang Ren (APS), Maria Chan (CNM), and Xianghui Xiao (APS)

Real-time imaging, scattering, and spectroscopy are primary experimental techniques to probe the dynamic process of materials formation, evolution, and structure-property relationships in energy materials.

In recent years, transmission electron microscopy (TEM) and synchrotron-based x-ray methods have been further developed to incorporate new environments and detectors to meet fast-growing research needs in energy materials science. Scientists have applied these techniques to investigate myriad processes, such as nanoparticle nucleation and growth and phase transitions of energy storage materials during the charge and discharge cycling. For example, *in situ* TEM provides a platform to study materials nucleation, growth, and electrochemically driven growth of dendrites on lithium anodes at the atomic scale.

In addition, the improvements in synchrotron x-ray optics and high-brightness coherent light sources allow investigation of phase changes and other chemical transformations with high spatial and temporal resolution.

Both electron and x-ray imaging methods provide opportunities to study energy materials in three-dimensional views, in addition to providing spectroscopic information.

Integration of these different techniques to study the energy materials in multimodal approaches at different length scales and temporal scales will help scientists further understand the working mechanisms of energy materials.

High-performance computation is crucial for the deployment of such multimodal imaging capabilities, both in the form of high-volume and high-speed data acquisition and processing and in the form of data interpretation via atomistic and first-principles simulations. The coupling of the experimental and computational development requires integrated teams and multidisciplinary efforts. Argonne National Laboratory has established the Integrated Imaging Initiative to emphasize the importance of multimodal imaging.

This workshop will cover the most recent developments and applications of *in situ* characterization techniques and high-performance computations for the study of energy materials. Particular attention will be paid to *in situ* multimodal imaging in the investigation of phase transformations, dynamics, crystal growth, crystal defect formation and elimination, chemical reactions, and interface dynamics in environmental conditions. The goal of this workshop is to provide a forum for experimental and computational scientists and facility users from various fields who are utilizing *in situ* methods to understand the structure, physics, and chemistry of energy materials.

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8:30 – 8:40 Opening Remarks

8:40 – 9:30 Nigel Browning (Pacific Northwest National Laboratory)
*Quantitative* in situ *Transmission Electron Microscopy*

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| 9:30 – 10:00 | Haimei Zheng (Lawrence Berkeley National Laboratory)<br>*Real-time Imaging of Materials Transformations in Liquids* |
| 10:00 – 10:35 | Break |
| 10:35 – 11:05 | Chongmin Wang (Pacific Northwest National Laboratory)<br>In situ *and* ex situ *TEM Study of Anode and Cathode for Lithium-ion Battery and Beyond* |
| 11:05 – 11:35 | Scott Warren (University of North Carolina at Chapel Hill)<br>*Identifying Champion Nanostructures with Electron, Light, and Force Microscopies* |
| 11:35 – 12:00 | Jeffrey Greeley (Purdue University)<br>*Computational Investigations of Interfacial Structure and Reactivity* |
| 12:00 – 1:30 | Lunch |
| 1:30 – 2:20 | Paul Fenter (Argonne National Laboratory)<br>In situ *Studies of Structures and Processes at Model Electrode/Electrolyte Interfaces* |
| 2:20 – 2:50 | Jiajun Wang (Brookhaven National Laboratory)<br>In situ *2D/3D Imaging of Battery Materials with Full-field Transmission X-ray Microscopy* |
| 2:50 – 3:20 | Break |
| 3:20 – 3:50 | Raymond Osborn (Argonne National Laboratory)<br>*Single Crystal Diffuse Scattering: Big Data beyond the Workflow* |
| 3:50 – 4:20 | Todd Turner (Air Force Research Laboratory)<br>*Microstructural Modeling of a Combined High-energy Diffraction Microscopy Experiment* |
| 4:20 – 4:50 | Mark Hereld (Argonne National Laboratory)<br>*An Integrated Platform for Studying Complex Biological Systems* |
| 4:50 | Closing Remarks |

WK7

## Quantitative *in situ* Transmission Electron Microscopy

**N.D. Browning[1,2], B.L. Mehdi[1,2], E. Jensen[1,2], P. Abellan[1], L.R. Parent[1,2], A. Stevens[3], D.A. Welch[4], R. Faller[4], J.E. Evans[5], C. Park[6], C.M. Wang[2,5], J.-G. Zhang[2,7], and K.T. Mueller[2,5,8]**

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[5] Environmental Molecular Sciences Laboratory, PNNL, Richland, WA 99352
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[7] Energy and Environmental Directorate, PNNL, Richland, WA 99352
[8] Department of Chemistry, Penn State University, University Park, PA 16802

Many processes in materials science, chemistry and biology take place in a liquid environment — such as the synthesis of nanoparticles, biological cellular functions and the operation of Li-ion/next generation batteries. In these cases, the overall process/function of the system is a result of a series of complicated transients, where a change in the order, magnitude or location of any of the individual steps can lead to a radically different result. Understanding and subsequently controlling the final system outcome can therefore be greatly aided by the ability to directly observe these fundamental transient processes as and where they happen. Aberration corrected (scanning) transmission electron microscopy [(S)TEM] has the spatial resolution (typically < 0.1 nm) to directly visualize the atomic scale structural and chemical variations taking place in materials. Historically, such high resolution microscopy has been used to analyze materials before and after a process takes place to infer the dynamics of what happened in between. While there are still great advances that can be made with such analyses (at the very least in providing benchmark structures for nanoscale systems), a major breakthrough in recent years has been the design and implementation of *in situ* gas and liquid stages that allow (S)TEM images to be obtained while the transient processes are actually taking place. Performing experiments using these *in situ* stages presents numerous challenges to the traditional means of analyzing samples in an electron microscope — we are now dealing with the variability of dynamic process rather than a more straightforward static structure. In this presentation, I will discuss the recent developments in the design and implementation of *in situ* stages being pursued at the Pacific Northwest National Laboratory (PNNL) that permit quantitative information to be extracted from the observations. Examples of the use of these capabilities for the direct imaging of the fundamental processes important for energy storage and conversion materials will be presented. As the *in situ* stages have been designed to be incorporated into both high spatial resolution aberration corrected (S)TEM and high temporal resolution Dynamic TEM (DTEM), the potential for future experiments to study fast dynamics, including those involving live biological structures, will also be discussed.

*This work was supported in part by the Joint Center for Energy Storage Research (JCESR), an Energy Innovation Hub funded by the Department of Energy, Office of Science, Basic Energy Sciences. The development of the stages was supported by the Chemical Imaging Initiative, a Laboratory Directed Research and Development Program at Pacific Northwest National Laboratory (PNNL). PNNL is a multi-program national laboratory operated by Battelle for the U.S. Department of Energy (DOE) under Contract DE-AC05-76RL01830. A portion of the research was performed using the Environmental Molecular Sciences Laboratory (EMSL), a national scientific user facility sponsored by the Department of Energy's Office of Biological and Environmental Research and located at PNNL.*

WK7

## Real-time Imaging of Materials Transformations in Liquids

**Haimei Zheng**

Materials Sciences Division, Lawrence Berkeley National Laboratory, Berkeley, CA 94720

An understanding of how materials grow and transform in their working environment is essential to the development of functional materials and efficient devices for energy applications. We study the growth and transformation of materials in liquids by the development and application of liquid cell transmission electron microcopy (TEM). In this talk, I will present our study of shape evolution mechanisms of colloidal nanoparticles using liquid cell TEM. There have been a lot of studies on controlling shape of nanoparticles since the catalytic or other surface-enhanced properties of the nanocatalysts are highly dependent on their shape. However, how facets develop during nanoparticle growth is largely unknown due to the lack of direct observation. Using *in situ* liquid cell TEM, we have been able to identify unique growth mechanisms and have discovered rules that are applied to bulk systems break down at the nanoscale. At the end, I will also briefly show our development of electrochemical liquid cells for the study of dissolution-deposition at the electrode-electrolyte interfaces in battery applications.

WK7

## *In situ* and *ex situ* TEM Study of Anode and Cathode for Lithium-ion Battery and Beyond

**Chong-Min Wang**

Environmental Molecular Sciences Laboratory, Pacific Northwest National Laboratory, Richland, WA 99354

Over the last decades, we have witnessed tremendous progress on the development of aberration corrected transmission electron microscopy and scanning transmission electron microscopy. As a result of this development, imaging of materials at atomic scale and spectroscopying at sub-nanometer scale become a routine practice. The questions now come to how we extend the microscopy and spectroscopy methodologies to analyze materials at or near realistic/operating condition, typically such as real-time observation of catalytic process, oxidation and reduction, bio-tissue in a liquid cell, defects generation and interaction under deformation conditions, mass transport and microstructural evolution, charge and ion transport process in electrochemical cells. In this presentation, I will focus on *in situ* TEM techniques that developed for probing into the structural and chemical information of energy storage materials. *In situ* high-resolution imaging enables direct observation of structural evolution, phase transformation and their correlation with mass, charge and electron transport, which provide insights as how active materials failure with cyclic charging and discharging of a battery. In perspective, challenges and possible direction for further development of the *in situ* TEM imaging and spectroscopic methods for energy storage materials and other field will also be discussed.

WK7

## Identifying Champion Nanostructures with Electron, Light, and Force Microscopies

**Scott C. Warren**

**Departments of Chemistry and of Applied Physical Sciences, University of North Carolina at Chapel Hill, Chapel Hill, NC 27599**

Assemblies of 0D, 1D, and 2D materials have emerged as one of the most important architectures for solar cells, fuel cells, batteries, and water-splitting devices. The complexity of these materials, as exemplified by the huge number of unique interfaces, has frustrated attempts to identify the relationships between structure and electronic properties, a knowledge of which are crucial for improving device performance. I describe the development of several techniques and instruments based on electron, light, and force microscopies that allow *ex situ* and *in situ* imaging of single nanostructures with a spatial resolution that is often below 2 nm. This presentation will focus on our recent efforts to correlate structure and properties in aggregates of nanoparticles that are of interest in solar water splitting. By performing correlations on statistically significant sample sizes, we are able to deduce how specific types of nanoparticle interfaces result in high performance in water splitting, ultimately allowing us to design macroscopic devices with record ("champion") performance. We will briefly describe how new microscopy techniques and instruments are allowing this approach to be generalized to new material systems.

WK7

## Computational Investigations of Interfacial Structure and Reactivity

**Jeff Greeley**

**Purdue University, School of Chemical Engineering, West Lafayette, IN 47907**

Advances in the theoretical understanding of interfacial electrocatalysis have, over the past decade, permitted the extension of periodic Density Functional Theory studies, which have traditionally been applied to probe chemistry at gas/solid interfaces, to electrochemical systems where potential-dependent reactions occur at liquid/solid interfaces. Indeed, such techniques have been employed to study a surprisingly wide class of chemical processes, ranging from electrochemical oxygen reduction to carbon dioxide reduction to water splitting. In this talk, I will briefly review the application of these simple techniques to the classic case of the hydrogen evolution reaction on transition metal alloys. Next, I will demonstrate how very similar approaches can be applied to understand certain aspects of reactivity at bifunctional oxide/metal interfaces, and I will close by drawing analogies between these electrocatalytic processes and traditional heterogeneous catalytic chemistries at metal/support interfaces.

WK7

## *In situ* Studies of Structures and Processes at Model Electrode/Electrolyte Interfaces

**Paul Fenter**

**Chemical Science and Engineering Division, Argonne National Laboratory, Argonne, IL 60439**

Understanding and controlling reactions within electrochemical energy storage systems is a significant scientific and technical challenge. This is due to the complexity of these systems (e.g., for both the solids and electrolytes), as well as the extreme environments and significant structural and chemical changes that can take place as a function of applied potential. The behavior at the solid-electrolyte interface itself is especially poorly understood. I will review our recent work in which we seek to isolate and understand the role of interfacial reactivity in these systems through *in situ*, real-time, observations of electrochemically driven reactions. This is achieved by observing well-defined model electrode-electrolyte interfaces using x-ray reflectivity. I will discuss two distinct types of electrochemical energy storage systems: 1) lithium ion battery chemistries in which energy is stored by lithium ion insertion into electrodes (e.g., Si, $Si_xCr$, Ge, NiO). The goal of this work is to control the complex lithiation reaction path of these conversion reactions through the use of thin-film and multilayer electrode structures; and 2) super-capacitor systems, in which energy is stored by surface adsorption. For these systems, we are studying the static structures and dynamical response of room temperature ionic liquids at potential-controlled carbon interfaces which we find have inherently slow dynamics associated with the reorganization of the interfacial RTIL structure.

*This work was supported as part of the Center for Electrochemical Energy Science (CEES) and the Fluid Interface Reactivity Structure and Transport Center (FIRST), which are Energy Frontier Research Centers funded by the U.S. Department of Energy, Office of Science, Basic Energy Sciences. The work was done in collaboration with T. Fister, S.S. Lee, A. Uysal, H. Zhou (ANL), J. Esbenshade, B. Long, A. Gewirth (UIUC), X. Chen, G. Evmenenko, M. Bedzyk (Northwestern), G. Feng, S. Li, P. Cummings (Vanderbilt), S. Dai (ORNL), and Y. Gogotsi (Drexel).*

WK7

## *In situ* 2D/3D Imaging of Battery Materials with Full-field Transmission X-ray Microscopy

**Jiajun Wang and Jun Wang**

**Photon Sciences Directorate, Brookhaven National Laboratory, Upton, NY 11973**

Electrochemically driven phase transformation directly influences electrode performance in lithium ion batteries. Advancing our understanding of the mechanism necessitates the development of advanced tools with *in situ* capability to track the dynamic phase and structural changes of battery materials at 2D and 3D. The synchrotron hard x-ray imaging technique is particularly interesting for applications in battery studies because of its natural characteristics: it is non-destructive, chemically and elementally sensitive, environmentally friendly, and highly penetrative to enable *in situ* study of a real battery[1–3]. Considerable progress in this field has been reported recently from our group, beamline X8C at the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory (BNL), where a new full-field hard x-ray imaging technique, transmission x-ray microscopy (TXM), has been developed and applied to battery microstructure study [4,5]. In this talk, we will present our recent work using *in situ*/*in operando* TXM approach to track phase transformation at 2D and 3D for anode (Tin) and cathode ($LiFePO_4$) battery materials [1–3]. Challenges and opportunities of TXM technology for energy materials research will be also discussed. This *in situ* imaging approach has a wide variety of applications in other fields, such as fuel cells, catalysis, environmental science and biological science.

[1] Wang, J., Chen-Wiegart, Y.K., and Wang, J. *In operando* tracking phase transformation evolution of lithium iron phosphate with hard x-ray microscopy. *Nat. Commun.* **5**, 4570 (2014).

[2] Wang, J., Chen-Wiegart, Y.K., and Wang, J. *In situ* three-dimensional synchrotron x-ray nanotomography of the (de)lithiation processes in tin anodes. *Angew. Chem. Int. Ed.* **126**, 4549–4553 (2014).

[3] Wang, J., Chen-Wiegart, Y.K., and Wang, J. *In situ* chemical mapping of a lithium-ion battery using full-field hard x-ray spectroscopic imaging. *Chem. Commun.* **49**, 6480–6482 (2013).

[4] Wang, J., et al., Automated markerless full field hard x-ray microscopic tomography at sub-50 nm 3-dimension spatial resolution. *Appl. Phys. Lett.* **100**, 143107 (2012).

[5] Wang, J., et al., Size-dependent surface phase change of lithium iron phosphate during carbon coating. *Nat. Commun.* **5**, 3145 (2014).

WK7

## Single Crystal Diffuse Scattering: Big Data beyond the Workflow

**Raymond Osborn**

Argonne National Laboratory, Argonne, IL 60439

Single crystal diffuse x-ray scattering is a three-dimensional probe of complex defect structures embedded in an otherwise perfect lattice, providing insight into the role of disorder in generating materials properties such as fast-ion conduction or relaxor ferroelectricity. With the new generation of fast area detectors, full three-dimensional volumes of reciprocal space can be generated using continuous sample rotations, with several thousand images collected in less than 10 minutes. We have been developing methods of handling data collected at rates of several GB per minute by streaming images to a data server, combining the frames in NeXus files that contain comprehensive experimental metadata, and transforming the arrays into reciprocal space for real-time inspection and manipulation. A collaboration with Argonne computational scientists is developing interactive tools for the data analysis of very large (10–50GB) remote data sets using resources such as Globus catalogs and the Swift parallel scripting language to enable flexible script-based approaches to 'playing' with the data once the basic data reduction workflow is complete. These tools are being written in a sufficiently general way that they may be useful in other scientific domains.

WK7

## Microstructural Modeling of a Combined High-energy Diffraction Microscopy Experiment

**Todd Turner**

Air Force Research Laboratory, Yellow Springs, OH 45387

The strategic development of new engineering materials with improved performance rests upon establishing computational models that link materials processing, microstructure, and properties/performance. However, validating micro mechanical models that capture the relevant deformation at the microstructural level has remained elusive without access to experimental data at the relevant grain-level scale. Emerging experimental techniques such as High Energy x-ray Diffraction Microscopy (HEDM) address a critical need with respect to validation experiments for models focused on the prediction of mechanical properties at the mesoscale, where the response of grains and similar microstructural features are explicitly tracked. This presentation will focus on work done at the 1-ID beamline at the Advanced Photon Source, where grain level morphology and elastic strain evolution were tracked through HEDM techniques. A crystal plasticity finite element model based on this experiment will be presented, and the results of the model will be compared to the experimentally obtained HEDM data.

WK7

## An Integrated Platform for Studying Complex Biological Systems

**Mark Hereld[1], Kenneth M. Kemner[2], Robin Lambert Graham[3], Gyorgy Babnigg[2], Frank R. Collart[2], Olliver Cossairt[4], Nicola J. Ferrier[1]Benjamin S. Glick[5], Philippe H. Noirot[2], Sarah L. O'Brien[2], Norbert F. Scherer[6], and Rosemarie Wilton[2]**

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[6]Department of Chemistry, University of Chicago, Chicago, IL 60637

Imaging is one of the most important and powerful methods yet devised to learn about the world around us. In the Small Worlds project, an interdisciplinary team of scientists at Argonne, University of Chicago, and Northwestern University are developing a new multi-modal imaging capability for studying complex multi-agent processes in cells and systems of cells across spatial and temporal scales. This experimental platform is composed of integrated hardware, software, and molecular-scale reporters that will enable the study of systems biology problems involving many parts and spanning spatial scales from the nanometer to the millimeter and temporal scales from subseconds to days.

We have begun to develop and integrate three imaging methods that, when used together, will lead to tremendous new understanding of the organization and dynamic function of a wide range of complex biological systems. The first approach, scanning x-ray fluorescence (XRF) imaging, allows sub-micron scale measurements of intact complex systems in their native environment, even if that environment is opaque to visible light. The second method, correlative electron-optical imaging, allows ultra-resolution imaging of whole organisms by transmission electron microscopy (TEM) and 3D spatial correlation with optical imaging of the identical (fixed) samples. The third proposed development, the novel 3D snapshot interferometric holographic microscope (3D-SIHM), is optical 3D microscopy of dynamic living systems with nanoscale resolution by interferometry, plus multi-scale volumetric imaging by holography in a "snapshot" mode for quantitative determination of transport on nanometer to 100 μm scales. The three approaches are synergistic when used in tandem with reporters that function across one or more of the imaging methods.

The contingent of capabilities developed in this project will enable construction of dynamic experiments that can track and correlate interrelated molecular actors in complex processes, while providing detailed corroboration and supplementary data across physical scales with qualitatively different imaging modalities.

# Wednesday, May 13
# Facility-specific Workshops

## APS Workshop 8
## *In situ* X-ray Characterization of Microstructure during Manufacture

Location: Bldg. 401, Room A5000

Organizers: Don Brown and John Carpenter (Los Alamos National Laboratory)

Recent years have seen tremendous advances in the ability to monitor materials microstructure, in both spatial and temporal resolution, at third-generation synchrotron x-ray sources. Concurrently, in an effort to rebuild manufacturing capability in the United States toward lower cost, more efficient production, "advanced manufacturing" techniques are being developed. Because of this timing, a marriage between manufacturing and characterization techniques is possible. Indeed, because of the particularly simple geometry of many of the characterization techniques, such as high-energy diffraction, small angle scattering, and radiography, it is natural to explore *in situ* x-ray characterization measurements during manufacturing.

The purpose of this workshop is to bring together both researchers who have already initiated *in situ* manufacturing programs and those who could benefit from such work. The intent is to draw scientists interested in either materials (including polymers, electronics, metals, and ceramics) or manufacturing processes (from traditional casting to simple heat treatments to the most modern advanced manufacture techniques) or both. The session will consist of several talks to sample the state of the field, followed by discussion of instrumentation needs (e.g., power or water needs) necessary to enable continued advancement of capabilities.

| | |
|---|---|
| 8:30 – 8:40 | Welcome & Introductory Remarks |
| 8:40 – 9:20 | Amy Clarke (Los Alamos National Laboratory)<br>*Multi-scale Prediction and Control of Metal Alloy Solidification Dynamics to Achieve Advanced Manufacturing* |
| 9:20 – 10:00 | Jon Emery (Argonne National Laboratory)<br>*A Portable, Modular Reactor for* in situ *Synchrotron X-ray Investigation of Atomic Layer Deposition Processes* |
| 10:00 – 10:30 | Break |
| 10:30 – 11:10 | Ruipeng Li (Cornell University)<br>*Evolution of Organic Thin Film Transistors Controlled by Blade-coating Method* |
| 11:10 – 11:50 | Aaron Stebner (Colorado School of Mines)<br>*A Novel* in situ *Planar Biaxial Experiment* |
| 11:50 | Wrap Up |

WK8

## Multi-scale Prediction and Control of Metal Alloy Solidification Dynamics to Achieve Advanced Manufacturing

**A.J. Clarke[1], D. Tourret[1], S.D. Imhoff[1], J.W. Gibbs[1], P.J. Gibbs[1], Y. Song[2], A. Karma[2], N.N. Carlson[1], K. Fezzaa[3], W.-K. Lee[4], pRad Team[1], D.R. Coughlin[1], J.K. Baldwin[1], and J.T. McKeown[5]**

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[2] Northeastern University, Boston, MA 02115
[3] Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439
[4] Brookhaven National Laboratory, Upton, NY 11973
[5] Lawrence Livermore National Laboratory, Livermore, CA 94550

Metal alloy solidification provides the first opportunity to affect structural, chemical, and defect evolution that dictates mechanical performance. Because solidification is multi-scale, we use state-of-the-art synchrotron x-ray and proton imaging to study solidification dynamics and inform advanced computational models. At the microscopic scale, we control thermal gradient and solid-liquid interface velocity during directional solidification to manipulate microstructural and chemical evolution. We quantitatively compare our x-ray imaging of a dilute aluminum-copper alloy with three-dimensional phase-field simulations. We are able to successfully predict microstructural characteristics with phase-field, but only if solutal convection — a key factor identified from our experiments — is incorporated. At the mesoscopic scale, the evolution of dendritic arrays in aluminum-based alloys is compared to dendritic needle network modeling. We also predict casting mold filling and local thermal gradients and solid-liquid interface velocities with continuum-scale process modeling to yield microstructural predictions within a casting. Our multi-scale integration of *in situ* characterization, modeling, and controlled processing will enable the prediction and control of microstructural evolution during solidification and advanced manufacturing of metal alloys.

*This work was supported by an Early Career award from the U.S. DOE, Office of Basic Energy Sciences, Division of Materials Sciences and Engineering.*

WK8

## A Portable, Modular Reactor for *in situ* Synchrotron X-ray Investigation of Atomic Layer Deposition Processes

**Jeffrey A. Klug[1], Matthew S. Weimer[1,2], Jonathan D. Emery[1], Angel Yanguas-Gil[3], Christian M. Schleputz[4], Sönke Seifert[4], Seth B. Darling[5] Mike J. Pellin[1], Jeffrey W. Elam[3], Alex B.F. Martinson[1], Adam S. Hock[2,6], and Thomas Proslier[1,7]**

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Atomic layer deposition (ALD) is a thin film growth technique that utilizes alternate self-limiting surface reactions of vapor-phase precursors. The self-limiting growth process afforded by ALD provides unique advantages that are unavailable through other vapor-phase deposition methods. Specifically, ALD excels at: 1) growth of highly uniform and conformal films on large area substrates or high aspect ratio nanostructures and 2) precise and accurate control over thin film coating thickness (sub-nanometer) and composition (atomic-scale). These characteristics are particularly applicable in, for example, manufacturing processes for microelectronics, catalysis, and solar energy conversion and storage. Ultimately, to best understand ALD processes, *in situ* monitoring of structural and chemical properties during deposition is required. This is particularly important during the early stages of deposition — a growth regime that critically influences the films' morphology, crystallinity, and composition. Furthermore, as ALD is increasingly applied in ultrathin film applications (< 10 nm), the early-cycle ALD nucleation behavior, which often deviates from linear, steady-state growth modes, begins to dominate film properties and has critical influence for device applications.

Synchrotron characterization techniques provide some of the most powerful tools for the study of film structure and chemistry. The brilliance and tunability of the source at the APS allow us to access scattering and spectroscopic techniques unavailable with in-house laboratory setups and provide us with the opportunity to probe various ALD processes *in situ* starting at the very first deposition cycle. Here, we present the design and implementation of a portable ALD reactor that possesses a modular reactor scheme that enables simple experimental switchover between various characterization techniques and beamlines. As first examples, we present *in situ* results for 1) x-ray surface scattering and reflectivity measurements of epitaxial ALD of ZnO on sapphire, 2) grazing-incidence small angle scattering of ALD-related sequential infiltration of trimethylaluminum and water into PS-*b*-PMMA block copolymer lamellar thin films, and 3) grazing-incidence x-ray absorption spectroscopy of nucleation-regime growth of $Er_2O_3$. Importantly, the *in situ* ALD reactor is designed to serve a broad user base with diverse experimental requirements and objectives, and is available for use to researchers upon request.

WK8

## Evolution of Organic Thin Film Transistors Controlled by Blade-coating Method

**Ruipeng Li and Detlef-M. Smilgies**

Cornell High Energy Synchrotron Source, Cornell University, Ithaca, NY 14850

Solution-processed organic thin film transistors (OTFTs) have attracted considerable attention due to their high performance, low cost and easy manufacture. Achieving high performance requires that the microstructure and morphology of solution-cast thin films — that develops via nucleation and growth processes — exhibit a high degree of crystallinity with two-dimensional in-plane π-stacking, a low density of grain/domain boundaries exhibiting low crystallographic misorientation. Efforts to tune the microstructures and morphologies of solution-cast thin films have been hampered by the lack of understanding and control over the nucleation and growth of the thin films as the molecules cystallize from the solution phase under highly non-equilibrium conditions.

Here, we demonstrate *in situ* characterization of the structural variation of the organic transistors cast by the doctor-blading coating process through microbeam Grazing Incidence Wide Angle X-ray Scattering (GIWAXS) at D-line in CHESS, Cornell University. The crystallization process is monitored for various coating speeds to control the evolution from anisotropic to isotropic films, which provides a full characterization of the nucleation evolution in the doctor-blading coating process. The results reveal the relationship between the microstructure, morphology and the device performance and direct fine tuning of coating parameters for future roll-to-roll processing of organic electronics devices.

WK8

## A Novel *in situ* Planar Biaxial Experiment

**Aaron Stebner**

Colorado School of Mines, Golden, CO 80401

Advanced alloys, such as lightweight metals and shape memory alloys, are becoming increasingly important to the advancement of many industries. They often possess complex microstructures that result in anisotropic and asymmetric behaviors, often due to twinning and phase transformation of low symmetry crystal structures. Because of this, their three-dimensional mechanical properties and mechanisms of deformation cannot be fully understood through uniaxial characterization and processing studies. To elucidate these behaviors, a custom planar biaxial load frame capable of *in situ* x-ray and neutron diffraction experimentation has been built. The instrument was designed to study any arbitrary plane-stress loading condition, in addition to load path change events. Thus, the micromechanics of full plane stress yield and transformation loci may be quantified in addition to path-dependent behaviors. We will review the new experimental capabilities, sample designs, and discuss implications for improving processing models of advanced alloys.

# Wednesday, May 13
# Facility-specific Workshops

## APS Workshop 9
## Workshop on Sn-119 Nuclear Resonant Scattering at the APS

Location: Bldg. 402, Room E1100/E1200

Organizers: Michael Hu and Bogdan Leu (APS)

The purpose of this workshop is to inform the broader general user community about the development of a new capability at Sector 30 regarding the use of the 119Sn nuclear resonance. The workshop will bring together experts in the field with potential new users to identify specific needs and develop plans to implement those requirements.

The nuclear resonant scattering studies performed at Sector 3 and Sector 16 in the last two decades have proved valuable in studying diverse materials, including porphyrins, proteins, enzymes and inorganic catalysts, superconductors, clathrates, thermoelectrics, metallic alloys, and earth-bound minerals under extreme pressures. Measurement of element-selective phonon density of states and extraction of numerous thermal and elastic constants motivated many new areas of research.

Similarly, measurement of nuclear forward scattering in the time domain with nanosecond resolution led to better understanding of electronic properties, such as valence and magnetism, in samples and under conditions that render these measurements impossible in the laboratory. For example, magnetism has been measured in monolayers or under pressures exceeding 1 Mbar.

Thus, the interest in nuclear resonant studies has grown to a point where it is necessary to optimize beamlines for different isotopes and to create more opportunities. One such opportunity presents itself at Sector 30. The working energy of the HERIX spectrometer at Sector 30 (23.7 keV) and the 119Sn nuclear resonance (23.88 keV) can be reached with the same monochromator. The combination of this cryogenically cooled, high-resolution monochromator with 1 meV resolution and a newly installed short-period undulator (1.72 cm) creates an ideal condition for such measurements. The available flux is better by almost an order of magnitude than at similar facilities around the world. Recent tests and scientific output [1] have demonstrated this capability at Sector 30.

Tin-based nuclear resonance studies are of interest to a variety of groups working with such materials as newly discovered superconductors, thermoelectric materials, clathrates, Sn-halides, porphyrins, and organometallic compounds. The combination of access to the 119Sn resonance with capabilities such as microfocusing for nanomaterial studies and infrastructure for high-pressure measurements should make the new technique highly attractive for new users.

Reference

[1] B.M. Leu et al., "Vibrational dynamics of the host framework in Sn clathrates," *Phys. Rev. B* **90**, 104304 (2014), DOI: http://dx.doi.org/10.1103/PhysRevB.90.104304.

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8:30 – 8:35 Welcome & Introductory Remarks

8:35 – 9:05 Ercan Alp (Argonne National Laboratory)
*Sn-based Nuclear Resonant Scattering Studies: Past and Present*

| | |
|---|---|
| 9:05 – 9:45 | John Tse (University of Saskatchewan)<br>*Chemical Bonding and Lattice Dynamics of Sn Compounds from 119-Sn Mossbauer* |
| 9:45 – 10:25 | Raphael Hermann (Forschungszentrum Juelich)<br>*Highly Anisotropic Lattice Dynamics in $[(SnSe)_{1.04}]_m$-$[MoSe_2]_n$ Ferrecrystals* |
| 10:25 – 10:35 | Break |
| 10:35 – 11:15 | Mathieu Roskosz (Univ. des Science et Tech. de Lille)<br>*Tin Isotopes: The Next Probe of Planetary Differentiation and Core Formation* |
| 11:15 – 11:55 | W. Robert Scheidt (University of Notre Dame)<br>*Tin Porphyrins 1969–2014* |
| 11:55 | Wrap-up and concluding remarks |

WK9

## Sn-based Nuclear Resonant Scattering Studies: Past and Present

**E.E. Alp, W. Bi, J. Zhao, M.Y. Hu, T.S.Toellner, and B. Leu**

Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439

$^{119}$Sn is the second most popular Mössbauer isotope after $^{57}$Fe. This is partly due to long lifetime of its parent isotope $^{119m}$Sn (250 days), relatively simple M1 transition from 3/2 to 1/2 state, reasonably long lifetime (18.3 nsec or 25.7 neV), and low transition energy (23.88 keV). Sn was among the first metals extracted (around 3500 BC). In modern times, Sn has found wider use in organotin compounds, transparent conductors, catalysis, protective coatings, float-glass production, and more recently in dye-sensitized solar cells, perovskites, Li-ion batteries, and tin chalcogenides as thermoelectric materials. Sn is also shown to be amenable to form two-dimensional topological insulators.

In this talk, I will introduce the Sn-based Mössbauer work done at the synchrotron radiation sources, their information content, and give a description of the new capability at Sector 30 beamline.

*This work is supported by U.S. Department of Energy (DOE), Office of Science, Basic Energy Sciences under contract DE-AC02-06CH11357.*

WK9

## Chemical Bonding and Lattice Dynamics of Sn compounds from 119-Sn Mossbauer

**John S. Tse**

University of Saskatchewan, Department of Physics and Engineering Physics, Saskatoon, Saskatchewan, Canada

In this presentation, I will present a general introduction on how 119-Sn Mossbauer spectroscopy can provide useful chemical information and lattice dynamics of Sn nuclei in a variety compounds. Special emphases will be on system that cannot be easily amenable to laboratory Mossbauer spectrometers, such as thin films and under extreme conditions.

WK9

## Highly Anisotropic Lattice Dynamics in $((SnSe)_{1.04})_m$-$(MoSe_2)_n$ Ferecrystals

**Raphaël P. Hermann**

Jülich Centre for Neutron Science, Forschungszentrum Jülich GmbH, 52425 Jülich, Germany
Materials Science and Technology Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831

The highly anisotropic lattice dynamics properties in SnSe based ferecrystals obtained from nuclear resonance inelastic x-ray scattering by the $^{119}$Sn Mössbauer resonance will be discussed. Ferecrystals and related misfit layered compounds exhibit extremely low lattice thermal conductivity. These materials multilayer materials exhibit turbostratic disorder between the two constitutent layers stacked in the direction perpendicular to the substrate, here *m* unit-cells of SnSe and *n* of $MoSe_2$. By measuring the density of phonon states for Sn with the beam in plane with the layers and perpendicular to the layers, highly contrasting speed of sound and force constants are found for the $(m,n)=(1,1)$ system. Further, a comparison of systems with $(m,n)=(1,1)$ and (4,1) reveals the importance of interface effects in determining the lattice dynamics properties. Overall the ferecrystals are ideal in revealing properties of phonons under confinement.

*Benedikt Klobes, Michael Hu, Matt Beekman, and David C. Johnson are acknowledged for the fruitful collaboration. The Advanced Photon Source at Argonne National Laboratory is acknowledged for provision of synchrotron radiation facilities at Sector 3.*

WK9

## Tin Isotopes: The Next Probe of Planetary Differentiation and Core Formation

**Mathieu Roskosz**

UMET, CNRS, University of Lille, France

Terrestrial planets (including Mars and the Moon) are differentiated into metallic cores and silicate mantles and crusts. This layered structure provides some of the most important properties of these planets such as a magnetic field that deflects the solar wind and plate tectonics. Understanding the thermodynamical conditions (P, T, $fO_2$) that prevailed during this differentiation is thus a major question in Earth sciences. The elemental concentrations of siderophile (iron-loving) elements in the Earth's mantle have been intensively studied. Based on this approach, it is now widely accepted that the upper mantle abundances have been set by metal-silicate equilibrium in an early magma ocean. However, the redox conditions and their evolution during planetary formation are still highly controversial.

A new approach of this question is based on the isotopic composition of siderophile elements (mainly Si and Fe). However, a quantitative analysis of data collected on natural samples requires knowing accurately the way isotopes partition between coexisting phases (iron-based metal alloys, silicates and sulfides in particular). The so-called *fractionation factor* can be determined experimentally but it requires drastic equilibrium conditions that are barely achievable at the high pressures and temperatures of interest. In the case of iron and tin these experimental difficulties can be overcome if fractionation factors are indirectly measured using a synchrotron-based inelastic spectroscopy. The determination of these factors is achieved by measuring phonon excitations using a kind of inelastic x-ray scattering based on special Mössbauer nuclei (NRIXS).

In this context the recent analytical developments make possible to analyze the isotopic compositions of tin in natural samples. So far, this system has never been successfully explored from a geochemical point of view. Though some attempts were made in the past, the chemical separation of tin from other elements present in rocks was a real issue that is being solved these days. Now, we have a unique opportunity to develop the tin isotope geochemistry. There are very few places in the world with sufficient x-ray intensity, proper high-resolution optics, and nanosecond time resolved detectors with high efficiency to measure $^{119}$Sn-specific properties of materials. The APS beamlines (Sector 3 and Sector 30) have developed proper crystal optics suitable for this purpose.

Here I will present known aspects of the physical and coordination chemistry of tin in geomaterials, how they should control tin isotopes fractionation and how Sector 30 will help us, in the coming years, to provide the mandatory fractionation factors to make Sn isotopes the new probe of the early evolution of the Moon, the Earth and Mars.

# Wednesday, May 13
# Facility-specific Workshops

## APS Workshop 10
## Application of Synchrotron X-ray Scattering Techniques to Nuclear Materials

Location: Bldg. 401, Room A5000

Organizers: Don Brown (Los Alamos National Laboratory) and Maria Okuniewski (Idaho National Laboratory)

The heyday of research into nuclear energy materials predated the modern model of the national user facility in the United States, which essentially was pioneered at Intense Pulsed Neutron Source at Argonne National Laboratory in the 1980s. As a result, many of the advanced characterization techniques that have been developed at neutron and synchrotron x-ray sources have not been brought to bear on nuclear materials.

The recent renewed interest in nuclear power, spurred by increased energy costs and concerns about climate change, has meant a resurgence in nuclear materials research, including work at user facilities. However, barriers to the study of naturally radioactive and activated materials still exist, in particular at synchrotron sources, which often do not have the infrastructure to deal with radioactive material that neutron sources inherently possess. The purpose of this proposed workshop is to gather those scientists currently utilizing the APS to study nuclear materials, as well as scientists whose programs could benefit from the advanced tools available at the APS.

The workshop will begin with several talks about cutting-edge nuclear material science to expose non-APS users to the possibilities for this type of work. It will conclude with a discussion of the current policies that govern the study of nuclear materials at the APS, including design of containment, shipping and local transport, sample handling, and completion of the experiment. Here again, participants who are not APS users will add valuable input as they can comment on best practices at universities and other government laboratories. The goal of this workshop is to produce a white paper compiling the expressed needs of the user community regarding radioactive sample handling at the APS and recommendations about how to accommodate those needs.

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| 1:30 – 1:40 | Welcome & Introductory Remarks |
| 1:40 – 2:10 | Jim Stubbins (University of Illinois Urbana-Champaign)<br>*Analysis of Nuclear Fuels, Cladding, and Structural Materials* |
| 2:10 – 2:40 | Meimei Li (Argonne National Laboratory)<br>*Synchrotron Radiation Characterization of Materials for Nuclear Energy Applications* |
| 2:40 – 3:10 | Mohamed Elbakhshwan (Brookhaven National Laboratory)<br>*Synchrotron X-ray Diffraction of Nuclear Materials* |
| 3:10 – 3:20 | Break |
| 3:30 – 3:50 | APS Radiation Safety Officer |
| 3:50 – 4:50 | Panel Discussion |
| 4:50 | Wrap Up |

WK10

## Analysis of Nuclear Fuels, Cladding and Structural Materials

**James Stubbins**

University of Illinois Urbana-Champaign, Urbana, IL 61801

The APS has provided substantial new understanding of the bulk effects that govern the performance of materials for nuclear applications. Much of this understanding is due to the fact that prior to very bright light sources, the performance of materials for nuclear application was determined on pre- and post-irradiated samples where only a "before and after" picture could be obtained. Current work with the APS has provided the opportunity to understand the active processes involved during the deformation of metallic alloys, which were previously not known. In particular, the role of various strengthening mechanisms was not fully characterized or appreciated. In this talk, the effects of various strengthening mechanism on nuclear materials performance will be discussed with particular attention to the new understanding of dynamic deformation processes.

WK10

## Synchrotron Radiation Characterization of Materials for Nuclear Energy Applications

**Meimei Li**

Argonne National Laboratory, Argonne, IL 60439

The use of synchrotron radiation offers new opportunities to advance the fundamental understanding of nuclear reactor materials in extreme conditions of irradiation, tempeature, stress and corrosion. This presentation will highlight recent studies using synchrotron x-ray scattering, spectroscopy and imaging techniques to understand the dynamic behavior of nuclear reactor materials and radiation damage over a wide range of lengh scales. The presentation will also discuss a rcently-developed new capability for *in situ* characterization of irradiated specimens under thermal-mechanical loading using combined techniques of far-field high-energy diffraction microscopy (ff-HEDM)/tomography/wide-angle x-ray scattering (WAXS)/small-angle x-ray scattering (SAXS). The containment design for safe handling of an irradiated specimen at an open beamline at the APS will also be discussed.

WK10

## Synchrotron X-ray Diffraction of Nuclear Materials

**Mohamed Elbakhshwan[1], David J. Sprouster[1], Simerjeet K. Gill[1], Sanjit Ghose[2], Eric Dooryhee[2], and Lynne E. Ecker[1]**

[1]Nuclear Science and Technology Department, Brookhaven National Laboratory, Upton, NY 11973
[2]National Synchrotron Light Source II, Brookhaven National Laboratory, Upton, NY 11973

Structural materials for nuclear energy systems are required to withstand radiation and corrosion at high pressure and temperature during reactor operation. Zirconium alloys used for cladding experience corrosion reactions with the coolant as well as the fuel during normal and accident conditions. Austenitic stainless steels, such as 304 and 347, are frequently used for core internals such as core barrels, bolts and support plates. Ferritic-martensitic steels such as T91 and HT9 are being considered as structural materials in advanced nuclear reactors. Exposure to radiation in a nuclear reactor can cause changes in the microstructure of the zirconium alloys or steels that can lead to brittle fracture, fatigue or irradiation-assisted stress-corrosion cracking. Radiation induced segregation can make the material prone to phase-transformations and the creation of new phases. In addition, the accumulation of defects (vacancy clusters) or helium can lead to void formation and swelling.

Synchrotron radiation sources can play an essential role in providing quantitative characterization of the atomic structure of complex materials; not just carefully prepared ideal systems, but materials that are actually used in nuclear reactors. The presentation will focus on the early oxidation states of fuel cladding alloys and the characterization of irradiated materials that was performed at the new state-of-the-art National Synchrotron Light Source II (NSLS II).

First, a sample environment was developed to perform *in situ* x-ray diffraction and x-ray fluorescence to understand the behavior of zirconium alloys under high pressure and temperature steam environment. Second, a robot has been developed at the X-ray Powder Diffraction (XPD) Beamline to automatically acquire high-resolution x-ray diffraction data, high energy (high Q) data for Pair Distribution Function Analysis, and small-angle x-ray scattering data for large number of samples of reactor pressure vessel steels or structural materials with varying chemistries, grain sizes, cold-work conditions and irradiation conditions. This tool provides safe, unmanned manipulation of relatively large numbers of radioactive samples for statistically representative, high-throughput measurements.

# Wednesday, May 13
# Facility-specific Workshops

## APS Workshop 11
## Tracking Electronic and Structural Dynamics in Proteins and Materials at Sector 14

Location: Bldg. 402, Room E1100/E1200

Organizers: Robert Henning (CARS, University of Chicago) and Anthony DiChiara (APS and CARS, University of Chicago)

The major upgrade to the BioCARS 14-ID beamline [1] produced unprecedented time-resolved capabilities that have been exploited for x-ray science spanning biology, chemistry, materials science, and physics. More recently, a new area detector (Rayonix MX340-HS, a 60-megapixel high-readout-rate integrating area detector) and secondary Kirkpatrick-Baez focusing mirrors were installed. The new equipment expands opportunities for time-resolved x-ray diffraction and solution scattering at 14-ID even further, by providing ~$15 \times 15$ μm$^2$ focused pink or monochromatic beam.

The workshop will bring together current practitioners and prospective new users of time-resolved pump-probe x-ray techniques from both biological and physical sciences. The goal of the workshop is to provide an overview of current capabilities and to discuss future challenges, needs, and opportunities, especially as they relate to brighter and smaller spots offered by the reduced-emittance APS MBA-lattice upgrade. The scientific emphasis will be on our new/current focal capability and the recent advances in serial crystallography, microcrystallography, x-ray excited warm dense matter, and time-resolved Laue diffraction and solution scattering.

Reference

[1] T. Graber et al., *J. Synch. Rad.* **18**, 658 (2011).

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1:30 – 1:40 Robert Henning (University of Chicago/BioCARS)
*Introduction*

1:40 – 2:10 Hyun Sun Cho (National Institutes of Health)
*Watching a Signaling Protein Function in Real Time via 150-picosecond Time-resolved Solution Scattering*

2:10 – 2:40 Xiaoshan Xu (University of Nebraska-Lincoln)
*Structural Dynamics in Improperly Multiferroic Hexagonal Ferrites*

2:40 – 3:10 Marius Schmidt
*Time-resolved Macromolecular Crystallography at the Synchrotron and at the X-ray FEL*

3:10 – 3:30 Break

3:30 – 4:00 Stephen Durbin (Purdue University)
*Interaction of X-ray and Laser Pulses in GaAs*

4:00 – 4:30 Sebastian Westenhoff (University of Gothenburg, Sweden)
*Signal Transduction in Phytochrome Photosensors Visualized by Time-resolved X-ray Scattering*

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| 4:30 – 5:00 | Aaron Lindenberg (Stanford University/SLAC)<br>*Ultrafast Studies of Interlayer Coupling in Transition Metal Dichalcogenide ReS2* |
| 5:00 | Concluding remarks |

WK11

## Watching a Signaling Protein Function in Real Time via 150-picosecond Time-resolved Solution Scattering

**Hyun Sun Cho, Friedrich Schotte, and Philip Anfinrud**

Laboratory of Chemical Physics, NIDDK, NIH, Bethesda, MD 20892

To understand how signaling proteins function, it is crucial to know the time-ordered sequence of events that lead to the signaling state. Using the time-resolved infrastructure we helped develop on the BioCARS beamline, we tracked the reversible photocycle of photoactive yellow protein following trans-to-cis photoisomerization of its p-coumaric acid (pCA) chromophore. Briefly, a picosecond laser pulse photoexcites pCA and triggers a structural change in the protein, which is probed with a suitably delayed picosecond x-ray pulse. When the protein is studied in a crystalline state, this "pump-probe" approach recovers time-resolved diffraction "snapshots" whose corresponding electron density maps can be stitched together into a real-time movie of the structural changes that ensue. However, the actual signaling state is not accessible in the crystalline state due to crystal packing constraints. This state is accessible in time-resolved small- and wide-angle x-ray scattering studies, which probe changes in the size, shape, and structure of the protein. The mechanistically detailed, near-atomic resolution description of the complete PYP photocycle developed from these studies provides a framework for understanding signal transduction in proteins, and for assessing and validating theoretical/computational approaches in protein biophysics. Thanks to an NIH-funded 2014 upgrade of the BioCARS beamline, the x-ray flux achievable and the rate at which images can be acquired has been boosted significantly, benefitting both time-resolved Laue crystallography and time-resolved SAXS/WAXS studies. This research was supported in part by the Intramural Research Program of the NIH, NIDDK.

WK-11

## Structural Dynamics in Improperly Multiferroic Hexagonal Ferrites

**Xiaoshan Xu**

University of Nebraska-Lincoln, Lincoln, NE 68588

Hexagonal ferrites, as a new family of multiferroic materials, exhibit ferromagnetism and ferroelectricity simultaneously. The improper ferroelectricity appears below 1050 K, driven by a non-polar structural distortion, while the weak ferromagnetism occurs below 130 K resulting from a competition between a couple of structural distortions. Since both the ferroelectricity and ferromagnetism have structural origins, a structurally mediated magnetoelectric coupling may be possible, as proposed by theory. As a key step, understanding the structural response to an electric field is crucial to understanding the magnetoelectric couplings in hexagonal ferrites. Using time-resolved x-ray diffraction, we probe the structural response to an effective electric field generated by the photo-induced charge carriers. By comparing the results with temperature-dependent structural refinements, dramatic responses of non-polar structural distortion are revealed, with the same time-scale of the charge carrier decay. The pattern of the lattice constants change indicates complex nature of the structural response. These results suggest that the structural response to the electric field may be a route for the magnetoelectric effect.

WK11

## Time-resolved Macromolecular Crystallography at the Synchrotron and at the X-ray FEL

**Marius Schmidt**

**UW-Milwaukee, Physics Department, Milwaukee, WI 53211**

Time-resolved crystallography unifies structure with kinetics. A time-series of difference maps is required to extract the molecular structures of reaction intermediates as well as the temporal evolution of the associated concentrations. Powerful synchrotron beamlines such as BioCARS at the Advanced Photon Source (APS) make the collection of these movies possible. *Multiple* time-series of photoactive yellow protein (PYP) can be collected rapidly. An additional experimental parameter such as the temperature can be varied. New, hitherto inaccessible, information can be extracted. Crystallography becomes five-dimensional [1,2]. At an x-ray free-electron laser, however, time-resolved serial femtosecond crystallography (TR-SFX) has several advantages: (i) due to the diffraction before destruction principle, radiation damage is negligible although enormous doses are deposited, (ii) small nano- and micro-crystals are utilized that can be easily and uniformly excited. Reactions in photo-reactive proteins such as PYP can be started by an optical laser pulse, and reactions in enzymes may be started by diffusion of substrate, (iii) cyclic reactions as the one in PYP and non-cyclic reaction such as those catalyzed by enzymes are conceptually on the same footing, (iv) the ultra-short, femtosecond x-ray pulses provide access to ultrafast time-scales beyond the pulse-limitations at the synchrotron. We present difference maps determined from TR-SFX [3] at beamline CXI at the LCLS. These results will pave the way to exciting, new experiments with photoreceptors and enzymes with serial crystallography at synchrotrons at the x-ray FELs.

[1] Schmidt, M., Srajer, V., Henning, R., Ihee, H., Purwar, N., Tenboer, J., and Tripathi, S., (2013), Protein energy landscapes determined by five-dimensional crystallography, *Acta Crystallogr D* **69**, 2534–2542.

[2] Schmidt, M., Graber, T., Henning, R., and Srajer, V., (2010), Five-dimensional crystallography, *Acta crystallographica. Section A, Foundations of crystallography* **66**, 198–206.

[3] Tenboer, J., Basu, S., Zatsepin, N., Pande, K., Milathianaki, D., Frank, M., Hunter, M., Boutet, S., Williams, G.J., Koglin, J.E., Oberthuer, D., Heymann, M., Kupitz, C., Conrad, C., Coe, J., Roy-Chowdhury, S., Weierstall, U., James, D., Wang, D., Grant, T., Barty, A., Yefanov, O., Scales, J., Gati, C., Seuring, C., Srajer, V., Henning, R., Schwander, P., Fromme, R., Ourmazd, A., Moffat, K., Van Thor, J.J., Spence, J.C., Fromme, P., Chapman, H.N., and Schmidt, M., (2014), Time-resolved serial crystallography captures high-resolution intermediates of photoactive yellow protein, *Science* **346**, 1242–1246.

WK11

## Interaction of X-ray and Laser Pulses in GaAs

**Stephen Durbin**

**Purdue University, Physics Building, West Lafayette, IN 47906**

Unusual excited states of matter can now be created with x-ray pulses at the APS Sector 14, a unique synchrotron source, creating energy densities comparable to optical laser pulses but with unique properties driven by the greater penetration depth and an energetic spectrum of electrons and fluorescence. An x-ray-induced excited state can be further excited by a laser pulse, or an x-ray pulse can excite a previously laser-pumped material. Combining the strikingly different interaction with materials of x-rays and optical photons can lead to new metastable states with unique dynamical properties, such as x-ray induced optical transparency. We report here on a double pump study of GaAs, using an x-ray pump with photons energetic enough to eject *K* electrons in GaAs, and an optical laser pump tuned for exciting valence electrons into the conduction band. Optical probes reveal that defect states surprisingly play a critical role in mediating strong interactions between the x-rays and the optical photons.

WK11

## Signal Transduction in Phytochrome Photosensors Visualized by Time-resolved X-ray Scattering

**A. Björling[1], H. Takala[1], O. Berntsson[1], S. Niebling[1], J.A. Ihalainen[2], and S. Westenhoff[1]**

[1]Department of Chemistry and Molecular Biology, University of Gothenburg, 40530 Gothenburg, Sweden
[2]Nanoscience Center, Department of Biological and Environmental Science, University of Jyväskylä, 40014 Jyväskylä, Finland

Sensory proteins must relay structural signals over large distances from the sensory site to regulatory output domains. Phytochromes are a major family of sensor proteins that control diverse cellular functions in plants, bacteria and fungi.

We study the structural dynamics of signal transduction in the photosensory core of the phytochrome form *Deinococcus radiodurans*. Our crystal and solution structures show an open and closed form of the dimeric protein for the activated and resting states, respectively [1]. This nanometer-scale rearrangement is controlled by refolding of an evolutionarily conserved 'arm', which is in contact with the chromophore. Time-resolved x-ray solution scattering data confirms that the opening movement is conserved in many bacterial phytochromes [2].

To arrive at these conclusions, x-ray crystallography was paired with time-resolved solution scattering. I will discuss two approaches to extract structural information from the latter data set. Firstly, structures are selected from many frames in unbiased molecular dynamics simulations [1,3]. Secondly, we have programmed a GROMACS-based molecular dynamics tool, in which the molecules are driven towards states that agree with the experimental scattering data [4]. I will discuss how these tools open up for studying the structural changes of proteins in solution.

[1] Takala, H. et al., Signal amplification and transduction in phytochrome photosensors. *Nature* **509**, 245–248 (2014).

[2] Björling, A. et al., Ubiquitous structural signalling in bacterial phytochromes. Unpublished.

[3] Arnlund, D. et al., Visualizing a protein quake with time-resolved x-ray scattering at a free-electron laser. *Nat. Methods* **11**, 923–926 (2014).

[4] Björling, A., Niebling, S., Marcellini, M., van der Spoel, D., and Westenhoff, S. Deciphering solution scattering data with experimentally guided MD simulations. *J. Chem. Theory Comput.* **11**, 150113180629009 (2015).

WK11

## Ultrafast Studies of Interlayer Coupling in Transition Metal Dichalcogenide ReS2

**Aaron M. Lindenberg**

Stanford University/SLAC, Stanford, CA 94305

I will describe recent experiments probing ultrafast structural dynamics in quasi-2D transition metal dichalcogenides. Time-resolved x-ray scattering measurements carried out at Sector 14 at the APS and at the Stanford Synchrotron Radiation Laboratory (SSRL) allow for direct probes of interlayer coupling phenomena in these materials, a key aspect of their functionality. Measurements reveal ultrafast acoustic responses and induced out-of-plane disorder following above-gap photo-excitation. If time allows, I will also describe recent complementary studies using time-resolved electron diffraction.

# 2015
# APS/CNM Users Meeting

# POSTER INDEX

## Advanced Photon Source

Use of the Advanced Photon Source, an Office of Science User Facility operated for the U.S. Department of Energy (DOE) Office of Science by Argonne National Laboratory, was supported by the U.S. DOE under Contract No. DE-AC02-06CH11357.

| Abstract | First Author | Title |
|---|---|---|
| **BIOLOGY** | | |
| A-1 | Becker | SONICC Implementation at GM/CA-beamline 23IDB at the APS |
| A-2 | Martynowycz | Cholesterol May Protect Mammalian Cells from Superwarfarins |
| **CHEMISTRY** | | |
| A-3 | Li | Study of Pressure/Temperature Effects on Borate Cross-linked Hydraulic Fracturing Fluids by Small Angle X-ray Scattering |
| A-4 | Liu | Exactly-doped Semiconductor Quantum Dots |
| A-5 | Moonshiram | Time Resolved X-ray Absorption Spectroscopy of a Highly Stable Nickel Catalyst for Artificial Photosynthesis |
| A-6 | Platero-Prats | *In situ* Pair Distribution Function Studies of Homogeneous Iridium Catalysts under Reaction Conditions |
| A-7 | Smirnova | *In situ* XAS Study of Oxygen-evolving Ruthenium Complex under Water Oxidation Conditions |
| A-61 | Fang | Pressure and Temperature Effects on the Antiferrodistortive Phase Transition and Phonon Softening in $SrTiO_3$ |
| **CONDENSED MATTER PHYSICS** | | |
| A-8 | Li | Strong Lattice Correlation and the Hopping of Non-equilibrium Hubbard Excitons in a Pseudospin-1/2 Mott Insulator $Sr_2IrO_4$ |
| A-9 | Wang | Magnetic Exchange Interaction between $Fe^{3+}$ and $Ho^{3+}$ Ions in Hexagonal $HoFeO_3$ Thin Films |
| **ENERGY AND GEOLOGY** | | |
| A-10 | Bolin | Iron Sulfide Particle Size Effects in a Maya Vacuum Resid and an Illinois #6 Argonne Premium Coal |
| A-11 | Hou | Hydration at Yttrium-stabilized Zirconia (110)-water Interface |

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| A-12 | Li | Uranium Immobilization by Plant Roots in Acidic Wetlands: Microcosms and XAFS Study |
| A-13 | Qiu | Structural Study of Surface Complexation of Pb(II) on a High-temperature Annealed Hematite (1-102) Surface |
| A-14 | Showalter | Sorption Mechanisms of Metals to Graphene Oxide |
| A-15 | Titi | X-ray Micro-computed Tomography for the Durability Characterization of Aggregate |

## HIGH PRESSURE

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| A-16 | Hrubiak | Technical Developments on High-pressure Melting of Metals Using Laser Pulse Heating and Synchrotron X-ray Diffraction |
| A-17 | Kono | X-ray Imaging for Studying Behavior of Liquids at High Pressures in Paris-Edinburgh Press |
| A-18 | Lin | Kinetics of the B1-B2 Phase Transition in KCl under Rapid Compression and Decompression |
| A-19 | Lin | Study of Phase Transition Pathways, Metastable Phases, Melting and Crystallization Using Time-resolved X-ray Diffraction and Dynamic (de)compression Techniques |
| A-20 | Park | Integration of Micro-x-ray Diffraction and X-ray Absorption Spectroscopy for High-pressure Research Using Dimaond Anvil Cells with Beryllium Gaskets |
| A-21 | Prakapenka | Combination of Laser Ultrasonics and Raman Spectroscopy in the Laser Heated Diamond Anvil Cell |
| A-22 | Smith | Fast Compression/Decompression of Materials in Diamond Anvil Cells |
| A-23 | Xiao | 16ID-D: High Pressure Spectroscopy Beamline at HPCAT |
| A-24 | Yu | A Paris-Edinburgh Cell for Liquid Silicate Structural Studies Using Monochromatic Diffraction and Multi-channel Collimator |

## INSTRUMENTATION

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| A-25 | Alkire | Checking Intensity Corrections on a CCD Detector with a Fiber-optic Taper |
| A-26 | Bradford | FASPAX: A Fast, Integrating Detector for the APS-upgrade |
| A-27 | Cummings | Elemental Sensitivity near the Atomic Scale |

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| A-28 | Fischetti | Minimizing Thermal Drift in a Dual Crystal Monochromator |
| A-29 | Macrander | Spherically Bent Quartz Analyzer Characterization at 1-BM |
| A-30 | Navrotski | Failure Modes of OFE and GlidCop® Copper Absorber Materials under X-ray Induced High Heat Load Thermal Fatigue Conditions |
| A-31 | Shi | Vibrations, Spot Size, and Flux Measurements at XSD Beamlines |
| A-32 | Saveliev | Vortex® SDD X-ray Spectrometers with Improved Counting Rate Performance |
| A-33 | Shirato | Low Temperature Synchrotron X-ray Scanning Tunneling Microscopy (LT-SXSTM) |
| A-34 | Shu | Mechanical Design of Multiple Fresnel Zone Plates Precision Alignment Apparatus for Hard X-ray Focusing in Twenty-nanometer Scale |
| A-35 | Stoupin | Rocking Curve Imaging at 1-BM X-ray Optics Test Beamline |
| A-36 | Woods | The APS Detector Pool |
| A-37 | Xu | New Mini-beam Collimator Provides Lower Background |
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## MATERIALS SCIENCE

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| A-40 | Croy | Understanding the Complex Structure of Composite, Lithium-ion Cathode Materials |
| A-41 | Nisbet | Ferroelectric Domain Engineered Strain Investigated Using Diffuse Multiple Scattering |
| A-42 | Moffitt | Probing the Role of Ga in Amorphous Conducting Oxides through Local Structure Studies |
| A-43 | Park | High-energy X-ray Techniques for Studying Nuclear Energy Relevant Materials |
| A-44 | Sen | *In situ* XAS Investigation of an Iron Oxide Battery Anode in Aqueous Electrolytes: Effect of Surface Modification on Rheology and Electrochemistry |

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| A-45 | Sun | Micro CT Microscopy and Ultra Small-angle Neutron Scattering Characterization of Alkali-Silica Reaction (ASR) Gel Development in Mortar Specimens with Recycled Glass Particles |
| A-46 | Weimer | Atomic Layer Deposition of Doped Films Studied by *in situ* Synchrotron Techniques with a Mobile and Modular Reactor |
| A-47 | Zeng | A Novel Phase in Li-Si System |

## NANOSCIENCE AND NANOTECHNOLOGY

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| A-48 | Burg | Metrology of Positioning Systems at the Nanometer Level |
| A-49 | Joyce | Enhancing Solar Cell Performance through the Use of Plasmon Generating Nanocomposite Materials |
| A-50 | Ulvestad | *Operando* Investigation of the Hydriding Phase Transformation in Single Palladium Nanocubes |
| A-51 | Werellapatha | X-ray Absorption Spectroscopy (XAS) Study of Sorption Mechanisms of Cd(II) to Hematite ($\alpha$-$Fe_2O_3$) Nano Particles with Varying Size and pH Environment |

## OTHER

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| A-52 | Blaiszik | Globus Data Publication Services and Their Application at the Advanced Photon Source |
| A-53 | Cardoza | Using Focused X-ray Pulses to Probe Microelectronic Devices for Single Event Effect Susceptibility |
| A-54 | Sharma | Swift Parallel Scripting for Fast, Productive Beamline Data Analysis |

## POLYMERS

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| A-55 | Huang | High Molecular Weight Insulating Polymers Can Improve the Performance of Molecular Solar Cells |
| A-56 | Iankovski | Dynamics of Nanoparticles within Gum Arabic Solutions |

## TECHNIQUE

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| A-57 | March | Tracking Molecular Dynamics from Picoseconds to Microseconds Using High-fidelity, Laser-pump, X-ray–probe Techniques |
| A-58 | Prescher | Dioptas and T-Rax: New Python-based Programs for On-the-fly 2D X-ray Diffraction and Optical Spectroscopy Data Analysis |

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| A-59 | Rivers | Pink Beam Tomography at 13-BM-D |
| A-60 | Randazzo | Probing Optically Dense, Flowing Systems with X-ray Absorption and Fluorescence of Argon and Krypton at Beamline 7BM |

## Center for Nanoscale Materials

Use of the Center for Nanoscale Materials was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.

### CHEMISTRY

| | | |
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| C-1 | May | X-ray Nanodiffraction Study of the Delithiation Mechanism of $LiFePO_4$ Single Particles |
| C-2 | Otto | Conjugated Bridge Effects on Triplet Generation in Ladder-type Perylene Dimers |

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| C-3 | Coenen | Preliminary Assessment of Microstructures on Siliceous Microfossils in Subglacial Lake Whillans, West Antarctica |

### MATERIALS SCIENCE

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| C-5 | Kinaci | Evolutionary Algorithm Search for Global Minimum Structures of Au Nano-clusters |
| C-6 | Narayanan | Bond Order Potential to Capture Size-dependent Dimensionality Effects in Au Nanoclusters |
| C-7 | Sen | $IrO_2$ Surface and Nanostructure Stability from First Principles and Variable Charge Force Field Calculations |

### NANOSCIENCE AND NANOTECHNOLOGY

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| C-8 | Cecil | New Fabrication Techniques for Cryogenic Micro-calorimeters |
| C-9 | Chen | Lithium Sulfur Batteries as Energy Storage Devices |
| C-10 | Humayun | ZnO Functionalization of Surface Pre-treated Multi-walled Carbon Nanotubes for Methane Sensing |

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| C-11 | Jiang | Nanostructure Electrodes for Perovskite Solar Cells and Electrochromic Devices |
| C-12 | Ocola | Sequential Infiltration Synthesized ZnO Nanostructures |
| C-13 | Ocola | Development Characteristics of Polymethyl Methacrylate in Alcohol/ Water Mixtures |
| C-14 | Poduri | Study of Polarization Anisotropy of Grown Cadmium Sulphide Nanowires |
| C-15 | Posada | Fabrication of Large Transition Edge Sensor Bolometer Arrays for CMB Measurements in the Upcoming SPT-3G Experiment |
| C-16 | Smith | Spectroscopic Imaging of NIR to Visible Upconversion from $NaYF_4$: $Yb^{3+}$, $Er^3$ Nanoparticles on Au Nano-cavity Arrays |
| C-17 | Spear | Frictional Properties of Graphene on Silica Surfaces with Nanoscale Roughness |
| C-18 | Wang | Novel Approaches towards Efficient Mg Rechargeable Batteries |
| C-19 | Wood | Protection of Exfoliated Black Phosphorus Transistors from Ambient Degradation |
| C-21 | Werellapatha | X-ray Absorption Spectroscopy (XAS) Study of Sorption Mechanisms of Cd(II) to Hematite ($\alpha$-$Fe_2O_3$) Nano Particles with Varying Size and pH Environment |
| C-22 | Wei | Integrated Photonics with Single-layer $MoS_2$ |

## TECHNIQUE

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| C-20 | Makarova | Microfabrication of All-copper Waveguides by UV-lithography |

## Exemplary Student Research Program

Using the world-class facilities at Argonne's Advanced Photon Source, area high school students and their teachers explore the principles and operation of these tools and conduct research during the school year. Under the guidance of staff scientists, each team develops an achievable project based on the techniques and limitations within a specific research group, prepares and submits a research proposal, sets up the experiment, gathers and analyzes their results, draws conclusions, and prepares a final poster for the Users Meeting.

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| ESRP-1 | Naperville Central High School | Nanoscale Elemental Fingerprinting of Historic Ink |
| ESRP-2 | Glenbard East High School | Sequestered Elements in Tissues of Water Willow and Freshwater Mussels as a Function of Sustainable Urban Stream Ecology |

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| ESRP-3 | Maplewood Richmond Heights High School | X-ray Crystallography of IF7 and Concanavalin-A |
| ESRP-4 | Downers South High School | The Effect of Increased Phosphorous on Metal Absorption in Lettuce |
| ESRP-5 | Neuqua Valley High School | Understanding the Reduction Mechanisms and Structural Changes of a Lithium-rich Oxide Material Using *Operando* X-ray Absorption Spectroscopy |
| ESRP-6 | Tinley Park High School | Determining the Suitability of Fly Ash Produced in Illinois for Use in Geopolymer Concrete |
| ESRP-7 | Oak Lawn Community High School | Comparing Extant and Fossilized Spiders to Determine Evolutionary and Preservational Changes Using Synchrotron X-ray Tomography |

# 2015
# APS/CNM Users Meeting

# APS POSTER ABSTRACTS

# Biology

A-1

## SONICC Implementation at GM/CA-beamline 23IDB at the APS

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Second order nonlinear optical imaging of chiral crystals (SONICC), based on femtosecond laser scanning microscopy, has been implemented at GM/CA@APS undulator beamline 23ID-B for rapid protein crystal localization and centering [1]. The technique is based on infrared laser light impinging on non-centrosymmetric crystals of proteins, which may selectively yield a frequency-doubled, visible signal generated by the anharmonic response of the electron cloud of the protein in response to the laser field. One aim of this method is to locate small crystals grown in opaque crystallization media for centering in x-ray beams of only a few microns or less in cross-section, such as for membrane-protein crystals grown in mesophase. The optical system implemented for generation and detection of Second Harmonic Generation (SHG) signals at beamline 23IDB has been described. Recent efforts towards providing user-friendly capabilities include: increasing rates of data acquisition, providing bright-field laser imaging capabilities, and incorporation of laser-safety interlocks suitable for a user program. SHG imaging has also enabled direct experimental visualization of electric fields generated by photoelectrons that are produced as a result of x-ray absorption, which provides insight on x-ray damage in samples [2]. Recent advances will be presented.

*GM/CA@APS has been funded in whole or in part with Federal funds from the National Cancer Institute (ACB-12002) and the National Institute of General Medical Sciences (AGM-12006). This research used resources of the Advanced Photon Source, a U.S. Department of Energy (DOE) Office of Science User Facility operated for the DOE Office of Science by Argonne National Laboratory under Contract No. DE-AC02-06CH11357.*

[1] J.T. Madden, S. Toth, C. Dettmar, J. Newman, R. Oglesbee, H. Hedderich, R. Everly, M. Becker, J. Ronau, S. Buchanan, V. Cherezov, M. Morrow, S. Xu, D. Ferguson, O. Makarov, C. Das, R. Fischetti, and G. Simpson. *J Synchrotron Rad.*, 2013, **20**, 531–540.

[2] C.M. Dettmar, J.A. Newman, S.J. Toth, M. Becker, R.F. Fischetti, and G.J. Simpson. *Proc. Nat. Acad. Sci.*, 2015, **112**, 696–701.

A-2

## Cholesterol May Protect Mammalian Cells from Superwarfarins

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Warfarin is the most widely used anticoagulant used in the world, and prescribed for the prevention of heart attack, strokes, and blood clots. Superwarfarins are modified analogues of the commonly used anti-coagulant warfarin. In contrast to warfarin, superwarfarins typically have 100-fold or greater potency to inhibit vitamin K production, and have an extremely long biological half-lives. The precise biophysical properties underlying those increases are not fully known, however the presence of aromatic rings make them strongly lipophilic which may contribute to increased tissue retention or binding to vitamin K epoxide reductase. We tested the possibility that superwarfarins exhibit strong binding interactions with membranes that are not observed with warfarin. Lipid monolayers comprised of DPPC (diphoshatidyl phosphocholine) or cholesterol were generated in Langmuir troughs, to which was introduced varying concentrations of the superwarfarin brodifacoum (BDF), or difenacoum (DiF), the des-bromo form of BDF. Interactions with the monolayers were probed by specular x-ray reflectivity and grazing incidence x-ray diffraction, and compared to results using warfarin. Reflectivity data reveals that both BDF and DIF interact with DPPC monolayers in dose-dependent manner at concentrations near their estimated lethal dose. In contrast, warfarin showed no interactions with DPPC. Grazing incidence x-ray diffraction shows a rearrangement of lateral crystalline order of the DPPC upon introduction of BDF and DiF. Neither BDF nor DiF showed significant interactions with cholesterol. Our *in vitro* studies show that BDF increases lactate production in rat glioma cells; however, following cholesterol depletion lactate production was significantly increased by BDF. These results demonstrate a preferential interaction of superwarfarins with zwitterionic phospholipids over cholesterol, suggesting a potential protective mechanism served by membrane cholesterol in mammalian cells.

# Chemistry

A-3

## Study of Pressure/Temperature Effects on Borate Cross-linked Hydraulic Fracturing Fluids by Small Angle X-ray Scattering

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Recently it was reported that borate cross-linked guar gums of the type that are commonly used in hydraulic fracturing fluids exhibit reversible pressure-induced changes in the viscosity that could potentially influence their application in the field under more adverse conditions, such as deeper wells. More recent work at SLB has demonstrated that the decrease in viscosity becomes evident at lower pressure as the temperature is increased. Similar effects were observed for polyvinyl alcohol (PVA) and a wide range of borate and polyborate cross-linking agents. We investigate these systems using *in situ* high-pressure/Temperature small angle x-ray scattering methods to obtain a better understanding of the underlying cause of the pressure effects on borate binding and polymer configuration under conditions of high temperature and pressure.

A-4

## Exactly-doped Semiconductor Quantum Dots

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Progress on the synthesis of colloidal semiconductor quantum dots (QDs) has largely focused on simple binary systems such as ubiquitous CdSe QDs. Recently, the introduction of guest impurities into such binary quantum confined systems has become topical. This is due to the fact that the incorporation of such impurities imparts a novel method for bandgap engineering beyond that achievable with size control. Unfortunately, present methods to synthesize doped QDs, such as the well-established rapid injection process, create samples with a dispersion of properties as the number of dopants per dot is dictated by Poissonian statistics. Lately, our group developed a strategy to synthesize QDs that contain an exact number of copper guest ions into CdSe [1], and recently into CdS and InP QDs matrixes controllably. This breakthrough has enabled us to fully characterize the structural, photophyscical, and chemical properties of those exactly-doped QDs systems. In this regard we have used Small Angle X-ray Scattering and X-ray Absorption Spectroscopy technologies. Specifically, we have demonstrated that the photophysics of copper dopants is strongly dependent on the host material in the case of Cu(I)-doped CdSe vs. CdS QDs. Another exciting result using time-resolved x-ray transient absorption spectroscopy has revealed the Cu(I) dopants embedded in CdS

QDs matrixes capture electrons after QD excitation, while the hole remains delocalized in the host. To the best of our knowledge, this is the first direct observation of Cu(I) dopants' role in the relaxation of excitation within a semiconductor host.

[1] A. Jawaid et al., *ACS Nano*, 2013, **7**, 3190–3197.

## A-5

## Time Resolved X-ray Absorption Spectroscopy of a Highly Stable Nickel Catalyst for Artificial Photosynthesis

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Production of cost-effective hydrogen gas through solar power is an important challenge of the Department of Energy among other global industry initiatives. In natural photosynthesis, the oxygen evolving complex (OEC) can carry out four-electron water splitting to hydrogen with an efficiency of around 60%. Although, much progress has been carried out in determining mechanistic pathways of the OEC, biomimetic approaches have not duplicated Nature's efficiency in function. Over the past years, we have witnessed progress in developments of light harvesting modules, so called chromophore/catalytic assemblies. In spite of reportedly high catalytic activity of these systems, quantum yields of hydrogen production are below 40% when using monochromatic light. Proper understanding of kinetics and bond making/breaking steps has to be achieved to improve efficiency of hydrogen evolution systems. This project shows the timing implementation of ultrafast x-ray absorption spectroscopy to visualize in "real time" the photo-induced kinetics accompanying a sequence of redox reactions in a nickel-based molecular photocatalytic system. Time-resolved XANES supplemented by Density Functional theory calculations illustrate formation of a Ni(I) species.

## A-6

## *In situ* Pair Distribution Function Studies of Homogeneous Iridium Catalysts under Reaction Conditions

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Homogeneous metal catalysis is a very common tool in organic synthesis. However, mechanistic aspects concerning many of these processes remain poorly understood. Knowledge of the metal local structure in solution and under catalytic conditions would allow the development of more efficient catalysts.

Iridium-based catalysts are versatile and very selective in a wide range of chemical transformations. We have reported a new Ir(III) complex containing an N-heterocyclic carbene ligand functionalized with an alcohol group (complex 1) that catalyzes the formation of carbon-nitrogen bonds with high efficiency [1]. The mechanism has been investigated experimentally (e.g., isotopic labeling and kinetic investigations) and with computational studies [2]. We have recently expanded this family of Ir-carbene catalysts by modifying the ligand (complexes 2 and 3), observing different catalytic activities depending on the metal coordination environments.

*In situ* Pair Distribution Function (PDF) analyses have been performed at 11-ID-B in solution. These measurements have provided critical insights the local structural changes of Ir-complexes under catalytic conditions.

[1] Bartoszewicz, A., Marcos, R., Sahoo, S., Inge, A.K., Zou, X., Martín-Matute, B., *Chemistry: A European Journal*, 2012, **18**, 14510.

[2] Bartoszewicz, A., Gonzalez Miera, G., Marcos, R., Norrby, P.-O., Martín-Matute, B., 2015, submitted.

## A-7

## *In situ* XAS Study of Oxygen-evolving Ruthenium Complex under Water Oxidation Conditions

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Today there is a strong need in using and storing energy from alternative sources. One of such possible sources is artificial photosynthesis (i.e., a process similar to the water oxidation that occurs during natural photosynthesis). Though several devices based on water splitting were created, their efficiency has to be improved. So far, ruthenium-based molecular catalysts are most stable and well studied. However, the behavior of ruthenium complexes during the process of water oxidation is not yet very clear. *In situ* studies under catalytic conditions may provide answers to the questions about process of water oxidation by ruthenium molecular catalysts and involved intermediates.

In this work, single site Ru complex $[RuII(bpy)(tpy)H2O]^{2+}$ (tpy = 2,2';6',2"-terpyridine, bpy = 2,2'-bipyridine) [1] was studied using *in situ* x-ray analysis under electrochemical conditions of water oxidation. Solution of Ru complex at different pH (pH=1 and pH=7) were electrolyzed at different voltages (1.6 and 1.8 V relative to normal hydrogen electrode) and x-ray absorption spectra were recorded during electrolysis. XAS measurements were done at beamlines 20-ID and 20-BM at Advanced Photon Source.

Applying 1.6 or 1.8 V potential to initiate water oxidation we quickly achieved sample oxidation and catalytic current. In spite of the claims that RuV=O species should be dominant at 1.8 V oxidizing potential [2], measured Ru oxidation state does not increase above RuIV while ligand environment of $[Ru(tpy)(bpy)H2O]^{2+}$ catalyst undergoes significant modification. After up to 18 hours of electrolysis, XANES comparison indicates that only Ru(IV) is presented in all solutions. Also, by comparing EXAFS spectra at different times during electrolysis, it was found that at pH=7 structure of Ru complex matches that of $[Ru{=}O(tpy)(bpy)]^{2+}$ and does not change with time. However, this is not the case at pH=1: there changes in first and second coordination spheres happen quickly after start of electrolysis. EXAFS and DFT modeling is ongoing to assign structure of new Ru(IV) intermediate. During electrolysis no new Ru-O-Ru bridge signatures were observed in EXAFS. Sample remains catalytically active. Clear solutions show no signs of sample degradation. X-ray damage analysis at Ru K-edge shown that inside electrochemical cell solutions of Ru complexes can be exposed for extended periods of time to 22–23 keV x-ray beam without causing changes to Ru oxidation state or ligand environment. This stability is due to low absorption of Ru K-edge x-rays by water and sufficient sample volume (5–10 ml) of the cell.

[1] Y. Pushkar, D. Moonshiram, V. Purohit, L. Yan, and I. Alperovich, *J. Am. Chem. Soc.* **136** (2014) 11938–11945.

[2] J.J. Concepcion, M.K. Tsai, J.T. Muckerman, and T.J. Meyer, *J. Am. Chem. Soc.* **132** (2010) 1545–1557.

## A-61

## Pressure and Temperature Effects on the Antiferrodistortive Phase Transition and Phonon Softening in $SrTiO_3$

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We report a study of the pressure-induced anitferrodistortive cubic-to-tetragonal phase transition in strontium titanate ($SrTiO_3$) at ambient temperature. High-resolution inelastic x-ray scattering measurements reveal the softening of a phonon mode ($R_{25}$) at the Brillouin zone boundary; a lattice distortion sets in at a critical pressure of 9.5 GPa, which corresponds to a critical volume reduction of 5.3 distortion can be induced under ambient pressure by lowering the sample temperature through a critical temperature of 105 K. The relationship between the two phase transitions is clarified by comparing the power laws of the pressure and temperature dependences of the softening behavior and by first-principles calculations of the energetics of the system.

# Condensed Matter Physics

A-8

## Strong Lattice Correlation and the Hopping of Non-equilibrium Hubbard Excitons in a Pseudospin-1/2 Mott Insulator $Sr_2IrO_4$

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In correlated oxides the coupling of quasiparticles to other degrees of freedom such as spin and lattice plays critical roles in the emergence of symmetry-breaking quantum ordered states such as high temperature superconductivity. Here we report a strong lattice coupling of excitonic quasiparticles in spin-orbital coupling Mott insulator $Sr_2IrO_4$ probed via optical photon excitation. Combining time-resolved x-ray diffraction and optical spectroscopy techniques, we further reconstructed spatiotemporal map of the diffusion of quasiparticles. In comparison with superconductor parent compound $La_2CuO_4$, the strong lattice correlation is unexpected due to the different electronic structure. The observation extends the similarity between $Sr_2IrO_4$ and cuprates to the strong coupling between lattice and quasiparticles which persists to highly non-equilibrium conditions.

A-9

## Magnetic Exchange Interaction between $Fe^{3+}$ and $Ho^{3+}$ Ions in Hexagonal $HoFeO_3$ Thin Films

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Multiferroic materials have been of great interest because the multiple ferroic orders simultaneously existing in these materials may lead to diversified applications in information technology, sensing, and actuation. However, multiferroic materials with room-temperature spin order are rare in nature. Engineered hexagonal $HoFeO_3$ (*h*-HFO) thin film is a promising candidate for a multiferroic material with room temperature ferromagnetism because of the expected exchange interaction between the $Ho^{3+}$ ions and $Fe^{3+}$ ions. Here we report the study of magnetic ordering and interactions in epitaxial *h*-HFO thin films by x-ray magnetic circular dichroism (XMCD) measurements. Epitaxial (0001) *h*-HFO thin films were deposited on (111) yttria-stabilized zirconia substrates via pulsed laser deposition. XMCD spectra were measured at the Fe $L_{2,3}$ edges and Ho $M_{4,5}$ edges at beamline 4ID-C of the Advanced Photon Source at Argonne National Laboratory. During the XMCD experiments, the magnetic field was applied parallel in the x-ray propagation direction and 60° away from the film normal (*c*-axis). Temperature dependence of the XMCD results show ferromagnetic ordering of $Fe^{3+}$ ions up to 200 K and paramagnetic behavior for $Ho^{3+}$ ions above 10 K. The saturation magnetic moment per $Fe^{3+}$ ion, calculated from the XMCD spectra using the sum rules, is 0.26 $\mu_B$, which is about 10 times larger than that in *h*-$LuFeO_3$ [1]. Magnetic moment per $Ho^{3+}$ is 3.02 $\mu_B$ measured at 10 K and 6 T magnetic field. The comparison of the XMCD polarities of the Fe $L_2$ peak and the Ho $M_5$ peak suggests that the average magnetic moments of $Fe^{3+}$ and $Ho^{3+}$ are aligned in the same direction. The observed enhancement of $Fe^{3+}$ moment can be attributed to the exchange interaction between the $Fe^{3+}$ and $Ho^{3+}$ ions.

*Work at Bryn Mawr College is supported by NSF CAREER award (No. 1053854) and work at Argonne National Laboratory is supported by U.S.-DOE, Office of Science, BES (No. DE-AC02-06CH11357).*

[1] Jarrett A. Moyer et al., *APL Materials* **2**, 012106 (2014).

# Energy and Geology

A-10

## Iron Sulfide Particle Size Effects in a Maya Vacuum Resid and an Illinois #6 Argonne Premium Coal

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Coal is, and will remain for the foreseeable future, a major source of energy worldwide. Despite advances in renewable and gas resources, the U.S. Energy Information Administration predicts that coal will provide the majority of fuel for the generation of electricity continuing up to at least 2035. An understanding of sulfur-based mineral behavior in coals is still needed for coal processing and mining.

The $FeS_2$ polymorph pyrite and marcasite were mixed together with a Maya Vacuum Resid coal model in known ratios, and a scheme was devised to deduce particle size. The behavior of these samples was then tracked with increasing pyrolysis. The results show that marcasite is much less structurally stable if the initial particle size is large. This will result in a sudden generation of $H_2S$ and generation of pyrrhotite, $Fe_{(1-x)}S$, which is a coal gasification catalyst.

A large-particled and a small-particled Illinois #6 coal Argonne Premium Coal was also pyrolized to various degrees. The iron sulfide behavior in this sample showed that there was a difference in transformations from pyrite to pyrrhotite and then to troilite dependent on the particle size. One explanation is the shrinking-core model, in which escaping sulfur gas eventually weakens the particles' outer sulfur-poor matrix, which breaks away, exposing the inner pyrite core.

A-11

## Hydration at Yttrium-stabilized Zirconia (110)-water Interface

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Interfacial structure of yttrium-stabilized zirconia or YSZ (110) in contact with de-ionized water under ambient condition has been investigated by high resolution specular x-ray reflectivity (HRXR). The interfacial electron density profile with atomic resolution is derived from a model structure factor analysis which reproduces the measured HRXR data. The derived interfacial structure indicates that a possibly re-adsorbed $Y^{3+}$ ion layer is sandwiched between surface oxygen layer and adsorbed water layer. The selective depletion among $Y^{3+}$ and $Zr^{4+}$ species is inferred based on their solubility in water. The re-adsorbed $Y^{3+}$ ions are suggested to be constrained from further propagating into the bulk aqueous phase by forming Y-O bonds with surface O atoms (i.e., forming an inner-sphere complex) and water molecules as well as by electrostatic interaction between the $Y^{3+}$ and the negatively charged vacancy sites of Y near the surface. The vacancy sites by $Y^{3+}$ depletion and inherent O vacancies on the surface are likely occupied by adsorbed water underneath the $Y^{3+}$ layer which overlaps with the surface oxygen layer. These results show characteristic interfacial hydration structure that compensates the depletion-induced surface charges by partially-hydrated species of the depleted ion itself. There is no indication of subsequent depletion of $Y^{3+}$ ions from deeper substrate within the experimental time scale (~8 hrs). While the majority of surface Zr atoms, exposed to water, seem to stay stable due to its intrinsically low solubility, the depletion of $Y^{3+}$ and its re-adsorption mechanism appears favorable in minimizing the interfacial energy.

## A-12
## Uranium Immobilization by Plant Roots in Acidic Wetlands: Microcosms and XAFS Study

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Uranium mobility and biogeochemistry is profoundly different in wetlands compared with upland sediments due to sharp geochemical gradients, elevated organic carbon concentrations and microbial activity, and the transient nature of hydraulic regimes. However, chemical speciation and biogeochemical behaviors of U in such systems are not well understood. At Savannah River Site (SRS), there were several former U processing facilities that released ~45,000 kg of depleted U into seepage ponds between 1958 and 1980. Approximate 70% of the depleted U still remains in the Tims Branch and associated wetlands, and becomes a sink of U contaminant to the environment. In this study, we investigated U chemical speciation and retention mechanisms by living plant roots in the acidic SRS wetlands using microcosms, U $L_3$-edge x-ray absorption fine structure (XAFS), and x-ray fluorescence (XRF) mapping.

The microcosm experiments were set up to mimic the biogeochemical conditions of the SRS wetlands. American bur-reeds (*Sparganium americanum*) transplanted from a noncontaminated SRS creek were grown in Ottawa sand pots under controlled temperature (25–30°C) and lighting (14 h/day). Several different nutrient solutions were supplied in sequence to keep the plants healthy while promoting Fe plaque formation on the roots and to allow the spiked U species to interact with plant roots under reducing conditions. The plant roots were then harvested, and together with the associated sand samples, examined using U $L_3$-edge XAFS and XRF mapping. Our findings were: 1) The U concentrations in the roots were qualitatively several hundred times higher than the U concentration in the Ottawa sands. 2) The chemical species of U on the Ottawa sands were U(VI). However, on 15 days after the root harvest, U chemical species on the plant roots were both U(IV) (up to 40%) and U(VI), and bound to carbon as a bidentate species and phosphorus as a monodentate species. 3) On 140 days after the root harvest, all U species became U(VI), but remained to bond with carbon as a bidentate species and phosphorus as a monodentate species. (4) XRF mappings indicated that there were hot spots for Fe and U, but they were not correlated in the distribution. The μ-XANES of Fe hot spots indicated the presence of Fe oxides and pyrite; while the chemical species of the U hot spots were U(VI), likely uranyl phosphate minerals as observed in electron scanning microscope (SEM) and the backscattering electron images of the roots. Together, these findings indicated that in the acidic SRS wetlands where U species is less likely to be immobilized by sediment minerals like goethite, they could be highly retained by plant roots that become a major source of high natural organic matters in the acidic SRS wetlands, even under oxidizing conditions.

## A-13
## Structural Study of Surface Complexation of Pb(II) on a High-temperature Annealed Hematite (1-102) Surface

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Structural study of Pb(II) adsorption at a high temperature-annealed hematite (1-102) surface was undertaken using crystal truncation rod (CTR) x-ray diffraction technique. The best fit CTR model suggests that Pb(II) adsorption at an annealed hematite (1-102) surface occurs under an inner-sphere mode with a bidentate edge-sharing binding configuration. The local structure of Pb surface complex species is a distorted trigonal pyramid with an average Pb-O bond length of 2.27 Å in good agreement with that reported in previous XAFS studies. Pb adsorption at three different bidentate sites (i.e., edge-sharing site of type 1, edge-sharing site of type 2 and corner-sharing site) was testified separately in the surface modeling. It was found that the edge-sharing site of type 2, which is the only site showing affinity for Pb(II) observed in the present study, is the preferable binding site mainly due to the existence of an O-Fe-O angle in this binding site structure more appropriate in producing a stable Pb surface complex species. In addition, it was also found that the Pb surface complex species bound at this site could be further stabilized via the formation of an extra weak Pb-O bond as well as a strong hydrogen bond between the distal oxygen and one surface oxygen. The bond valence rule was employed as an extra constraint in the process of CTR modeling to assure the steric feasibility in the optimized surface complexation structure. The post-model bond valence analysis suggests that both surface atoms and sorbate atoms in the best fit model structure could achieve a bond valence saturation state after a proper assignment of protonation schemes to under-coordinated oxygen groups. An unique stoichiometry of Pb(II) adsorption on the hematite (1-102) surface was proposed based on the best fit surface complexation structure as well as the protonation schemes obtained in the bond valence analysis, and the proposed stoichiometry indicates that the source of proton releasing is solely through the deprotonation of surface functional groups, which are chemically bound to Pb atom in the process of surface complexation. The molecular-scale structural information derived from this study is a great advance in the understanding of the Pb adsorption on a hematite (1-102) surface, which can be applied for a better modeling of the environmental behavior of Pb(II).

A-14

## Sorption Mechanisms of Metals to Graphene Oxide

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Environmental toxic metal contamination remediation and prevention is an ongoing issue. Graphene oxide is a promising material that adsorbs significant quantities of a wide variety of heavy metals under different pH and ionic strength conditions. We present x-ray absorption fine structure (XAFS) spectroscopy results investigating the binding environment of Pb(II), Cd(II), and U(VI) ions onto multi-layered graphene oxide (MLGO). Analysis indicates that the dominant sorption mechanism of Pb and U to MLGO changes as a function of pH. In contrast, the sorption mechanism of Cd to MLGO remains electrostatic across a wide range of pH values for a variety of pH values and ionic strength conditions. Determination of the sorption mechanism of various metals to MLGO will guide remediation strategies and prevention methods for toxic metal removal from aqueous environments.

A-15

## X-ray Micro-computed Tomography for the Durability Characterization of Aggregate

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A novel method, x-ray computed tomography, has recently emerged as a powerful, non-destructive methodology for material characterization, including geomaterials. This method produces 3D images of the object that can be analyzed in various ways based on the purpose of the scan. The objective of this research is to use x-ray CT technology to investigate the internal structure and porosity of various types of aggregates such as limestone, granite, and quartzite. In addition, this research used x-ray CT technology to investigate the influence of harsh environments such as freezing and thawing on the durability of the aggregate. Virgin and treated aggregate specimens were subjected to x-ray CT to obtain high-resolution 3D images. Aggregate treatments (wetting/drying and freeze-thaw cycles) were conducted using the sodium sulfate soundness test and the actual free-thaw test. The CT scans were carried out using a sector 13-BMD synchrotron microtomography beamline at the Advanced Photon Source of the Argonne National Laboratory, Illinois. Analysis was conducted on the acquired 3D high-resolution images to investigate the pore structure and micro-cracks of these aggregates types. The x-ray CT technology was useful for visualizing the internal structure of aggregate particles with high resolution. This visual inspection provided information on pore space characteristics such as pore shape, connectivity, and distribution. In addition, volumetric quantities such as the volume of aggregate particles and the volume of pore space were identified and measured. These measured quantities were used to calculate porosities of the investigated aggregates, which provided properties of these aggregates using the constructed 3D CT images (non-conventional method). Sodium sulfate soundness test effects on the treated aggregates (degradation, disintegration, and weathering) were significant, as observed in the 3D CT images of treated aggregate particles. Pore space volume increased as the aggregate particles were treated with wetting/drying cycles of sodium sulfate solution. The sodium sulfate soundness test significantly affected the permeable (connected) pore space and induced degradation/disintegration, which increased the volume of connected pore space with the increase of the number of wetting/drying test cycles. Isolated pore space remained unchanged with the number of sodium sulfate test cycles, since the salt could not penetrate these pores to induce internal force of expansion, which degraded and disintegrated the aggregate structure. The freeze-thaw test induced changes to the pore space of the treated aggregates with a noticeable impact on the connected pore space of the aggregate particle.

# High Pressure

A-16

## Technical Developments on High-pressure Melting of Metals Using Laser Pulse Heating and Synchrotron X-ray Diffraction

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We discuss several important experimental developments relevant to carrying out careful structural measurements on pure metals close to and above their melting temperature with pulse laser heated diamond anvil cell (DAC) and synchronized *in situ* x-ray diffraction. Precise high temperature generation and measurement for many metals close to their melting temperatures is not feasible with continuous laser heating in a diamond anvil cell approach due to the chaotic nature of the temperature response of the sample as well as chemical reactions which can take place in a DAC over even a relatively short time scale. However, provided that a number of important experimental considerations have been met, metal samples in a diamond anvil cell can now be controllably laser heated to 6000 K for relatively long pulse periods of 10–30 milliseconds, which is enough time to simultaneously collect x-ray diffraction in a single pulse with the currently available x-ray photon flux at the beamline 16-IDB of Advanced Photon Source. Time-scale dependent temperature response of the sample during the laser pulse duration is one of the most critical experimental variables and several key strategies for running successful experiments are presented. Several developments in areas such as sample preparation, sample chemistry during laser heating, laser pulse time-structure, alignment and synchronization of laser pulse, the temperature measurement and the x-ray beam will be discussed. We also present an example of an experiment involving controlled heating and measurement of x-ray diffraction of molybdenum metal samples under high pressure to temperatures thousands degrees above the melting point.

## A-17

# X-ray Imaging for Studying Behavior of Liquids at High Pressures in Paris-Edinburgh Press

**Yoshio Kono[1], Curtis Kenney-Benson[1], Changyong Park[1], Yanbin Wang[2], and Guoyin Shen[1]**

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Several x-ray techniques for studying structure, elastic properties, viscosity and immiscibility of liquids at high pressures have been integrated using a Paris-Edinburgh press at the 16-BM-B beamline of the Advanced Photon Source [1]. Here we report the development of x-ray imaging techniques suitable for studying behavior of liquids at high pressures and high temperatures. White x-ray radiography allows for imaging phase separation and immiscibility of melts at high pressures, identified not only by density contrast but also by phase contrast imaging in particular for low density contrast liquids such as silicate and carbonate melts. In addition, ultrafast x-ray imaging, at frame rates up to ~$10^5$ frames/second (fps) in air and up to ~$10^4$ fps in Paris-Edinburgh press, enables us to investigate dynamics of liquids at high pressures. Very low viscosities of melts similar to that of water can be reliably measured [2–4]. These high-pressure x-ray imaging techniques provide useful tools for understanding behavior of liquids or melts at high pressures and high temperatures.

[1] Y. Kono, C. Park, C. Kenney-Benson, G. Shen and Y. Wang, *Physics of the Earth and Planetary Interiors* **228**, 269–280 (2014).

[2] Y. Kono, C. Kenney-Benson, C. Park, G. Shen and Y. Wang, *Physical Review B* **87** (2), 024302 (2013).

[3] Y. Kono, C. Kenney-Benson, D. Hummer, H. Ohfuji, C. Park, G. Shen, Y. Wang, A. Kavner and C.E. Manning, *Nature Communications* **5**, 5091 (2014).

[4] Y. Kono, C. Kenney-Benson, Y. Shibazaki, C. Park, G. Shen and Y. Wang, *Physics of the Earth and Planetary Interiors* **241**, 57–64 (2015).

## A-18

# Kinetics of the B1-B2 Phase Transition in KCl under Rapid Compression and Decompression

**Chuanlong Lin, Jesse Smith, Stanislav Sinogeikin, Changyong Park, Yoshio Kono, Curtis Kenney-Benson, Eric Rod, and Guoyin Shen**

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Kinetics of the B1-B2 phase transition in KCl has been investigated at different (de)compression rates in a dynamic diamond anvil cell using time-resolved x-ray diffraction. The pressure (time)-dependent x-ray diffraction images show that the grain size of the product phase in the B1-B2 phase transition is (de)compression-rate dependent, *viz.*, slow (de)compression may result in large grasins and rapid (de)compression may result in small grains. The over-(de)pressurization increases with (de)compression rates. There are two stages for the increase of over-(de) pressurization: one is a rapid increase at low compression rate and the other is slow increase at high compression rate. The logarithm of the transition time, defined by the coexistence of B1 and B2 phases, has a linear relationship with the logarithm of the compression rates which, when extrapolated this linear relationship to high compression rates, is in agreement with shock-wave data. The volume fraction as a function of pressure shows sigmoidal curves against pressure at different (de)compression rates. Based upon classical nucleation and growth theories (Johnson-Mehl-Avrami-Kolmogorov theories), we propose a model to fit the experimental data of the volume fraction as a function of pressure. Analogous to temperature-induced phase transitions at isothermal heating or non-isothermal heating conditions, we define an effective activation energy ($Q_{eff}$) that can be experimentally determined by fitting the (de)compression-rate dependent volume fraction. It is found that the change of the nucleation mechanism, transition time, nucleation rate, and grain growth rates are related to compression-rate dependent effective activation energy. Our dynamic (de)compression experiments on KCl may bridge a gap between static compression and shock-induced phase transition.

## A-19

## Study of Phase Transition Pathways, Metastable Phases, Melting and Crystallization Using Time-resolved X-ray Diffraction and Dynamic (de)compression Techniques

**Chuanlong Lin, Jesse Smith, Eric Rod, Stas Sinogeikin, and Guoyin Shen**

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High-pressure Diamond Anvil Cell–based time-resolved x-ray diffraction and dynamic (de)compression techniques at High Pressure Collaborative Access Team (HPCAT) provide the possibility to observe (de)compression-rate dependence of phase transition pathways, pressure-quenching of metastable phases, as well as kinetics of phase transformations, melting and crystallization. Here we present some examples of our recent experimental results of dynamic (de)compression in common elements and compounds — water, silicon and gallium.

For water, we observed compression-rate dependence of phase transition pathways directly using time-resolved x-ray diffraction. We found that at large compression rates water transformed to ice VII directly without going through ice VI, while at slower compression rates it crystallizes to ice VI phase first and only then to ice VII.

It is well known that silicon has several metastable at low pressures. We consistently observed different metastable phases (crystalline and amorphous) or mixture of them at different controlled decompression rates, and these metastable phases can be kept for a long time at ambient conditions.

In pure metal gallium we studied the process of cyclic melting and crystallization under cyclic decompression and compression. We found that the compression rate has a remarkable effect on the phase transition/crystallization (over-pressurization), while during decompression the rate has a negligible effect on transition/melting pressure (over-depressurization).

In summary, the combination of time-resolved XRD and dynamic (de)compression techniques is a powerful tool for studying the rate dependence of phase transition pathways, metastable phases, and process of melting and crystallization in the high-pressure field. These techniques are currently available at HPCAT.

## A-20

## Integration of Micro-x-ray Diffraction and X-ray Absorption Spectroscopy for High-pressure Research Using Dimaond Anvil Cells with Beryllium Gaskets

**Changyong Park, Curtis Kenney-Benson, Eric Rod, Ligang Bai, and Guoyin Shen**

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X-ray absorption spectroscopy (XAS) measurements using diamond anvil cells suffer from "diamond glitches," when the absorption spectra are measured with transmitted x-rays through the anvils. Efforts have been made to remove glitches in recent years (e.g., using nano-diamond anvils, by rotating the sample, or using focused beam with a polycapillary). Here we introduce a method for bypassing the problem by utilizing an x-ray transparent beryllium gasket in radial transmission geometry (i.e., x-rays pass through the beryllium gasket perpendicular to the loading axis). The beryllium gasket technique is well-used in high-pressure radial diffraction experiments. The versatile setup of the HPCAT 16-BM-D beamline at the Advanced Photon Source, which can adapt 90-degree rotation of sample stage with less than 1 μm precision, allows a combined transmission XAS measurement, utilizing a diamond anvil cell with beryllium gasket, with a typical micro-XRD experiment. The fixed exit feature of the monochromator together with achromatic KB focusing mirrors facilitates a wide range of energy change for both XRD and XAS without significant changes in beam profile and intensity. This further allows back-to-back switching between XRD and XAS measurements at an identical sample condition. A study of phase transition behavior in an isostructural volume collapse system, $PrH_2$, has been performed with the combined XRD-XAS. The XRD was measured at 36.000 keV and the XAS was measured at the K-edge of Pr around 42 keV for each studied pressure point. The experimental results are demonstrated and discussed in terms of direction toward future development for the broader application.

A-21

## Combination of Laser Ultrasonics and Raman Spectroscopy in the Laser Heated Diamond Anvil Cell

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Understanding of the elastic behavior of minerals under high pressure is a crucial factor for developing a model of the Earth structure since the information about Earth interior comes mainly from seismological data. Laboratory measurements of velocities and other elastic properties of minerals are the key for understanding observed seismic information, allowing us to translate it into quantities such as chemical composition, mineralogy, temperature, and preferred orientation of minerals. Laser ultrasonics (LU) combined with diamond anvil cell (DAC) demonstrated to be an appropriate technique for direct determination of the acoustical properties of solids under high pressure. The use of lasers generating subnanosecond acoustical pulses in solids allows measurements of the velocities of shear and longitudinal waves propagated in opaque materials *in situ* at high pressure and temperature [1–4]. Absorption of the incident laser pulse energy and the associated temperature gradients induces a rapidly changing strain field. This strain field, in turn, radiates energy as elastic (ultrasonic) waves. At low pulse power, this is an entirely thermoelastic process resulting in no damage to the sample. The acoustic echo arriving at the probed surface causes both the displacement of the surface (a few nanometers) and the strain in the subsurface material, which might be detected through its influence on the optical reflectivity of the material (i.e., through the acousto-optic effect).

The details of the advanced system including laser ultrasonics in a point-source-point-receiver configuration coupled with Raman spectroscopy and laser heating techniques in the DAC for studying elastic properties of materials *in situ* at high pressure and temperature will be demonstrated. Future combination of this innovative system with high-resolution synchrotron x-ray micro-diffraction technique at GSECARS (Sector 13, APS) for full characterization of materials at extreme conditions will be discussed.

[1] N. Chigarev, P. Zinin, L.C. Ming, G Amulele, A. Bulou, and V. Gusev, *Appl. Phys. Lett.* **93,** 181905 (2008).

[2] N. Chigarev, P. Zinin, D. Mounier, A. Bulou, L.C. Ming, T. Acosta, and V. Gusev, *High Pres. Res*. **30**, 78 (2010).

[3] P.V. Zinin, V. Prakapenka, K. Burgess, and D.Y Velikovsky, *Scientific Notes of the Physics Department of Moscow State University* **6,** 146314 (2014).

[4] K. Burgess, V. Prakapenka, E. Hellebrand, and P.V. Zinin, *Ultrasonics* **54**, 963 (2014).

A-22

## Fast Compression/Decompression of Materials in Diamond Anvil Cells

**Jesse Smith, Chuanlong Lin, Ligang Bai, Eric Rod, Stanislav Sinogeikin, and Guoyin Shen**

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Fast compression/decompression constitutes one of the fundamental technical approaches to time-resolved high pressure research in the diamond anvil cell. Through careful choice of pressure generating apparatus and relevant time scale, a number of important scientific challenges can be address including, for example, non-equilibrium transformations and phase boundaries, unusual thermodynamic pathways to metastable phases, and compression-dependent nucleation rates and crystal growth. In this work we show preliminary results from a number of different scientific studies, each taking advantage of a particular apparatus and compression rate to measure, for example, equations of state at ambient and elevated temperatures, high sample compression and strain rates in materials, and compression-rate dependent sample stress and subsequent relaxation.

A-23

## 16ID-D: High Pressure Spectroscopy Beamline at HPCAT

**Yuming Xiao, Paul Chow, Genevieve Boman, Ligang Bai, Eric Rod Arunkamar Bommannavar, Curtis Kenney-Benson, Stanislav Sinogeikin, and Guoyin Shen**

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As part of a third generation synchrotron radiation beamline dedicated to high pressure research, 16-IDD of HPCAT at the Advanced Photon Source engages in x-ray spectroscopy research of samples under high pressures, typically in diamond anvil cells (DAC) [1].

The spectroscopy line consists of IDA with a liquid nitrogen cooled Si (1 1 1) double crystal monochromator with 4.5–37 keV energy range, transport lines; IDC with an interchangeable high-resolution monochromator at ~2 meV energy resolution and two 1-meter K-B mirrors; and IDD, the experiment station, with a 2.7 meter 0–90 degree horizontal inelastic x-ray scattering spectrometer and a dedicated x-ray emission spectroscopy setup. Typical beam size at sample position is ~25 (V) × 50 (H) $\mu m^2$ at FWHM when using meter-long KB mirrors, smaller beam size (4 × 5 $\mu m^2$) can be achieved by using a pair of 200 mm KB mirrors.

Current techniques include x-ray emission spectroscopy, inelastic x-ray scattering and nuclear resonant scattering. Examples of high pressure studies using these techniques and recent developments such as 7-element analyzer array for XES will be discussed in details in the meeting.

[1] Shen, G.Y.; Chow, P.; Xiao, Y.M.; Sinogeikin, S.; Meng, Y.; Yang, W.G.; Liermann, H.P.; Shebanova, O.; Rod, E.; Bommannavar, A.; and Mao, H.K., *High Pressure Research*, 2008, **28**, 145–162.

A-24

## A Paris-Edinburgh Cell for Liquid Silicate Structural Studies Using Monochromatic Diffraction and Multi-channel Collimator

**Tony Yu[1], Yanbin Wang[1], Clemens Prescher[1], Vitali Prakapenka[1], Lawrie Skinner[2] , Peter Eng[1], Joanne Stubbs[1], Yoshio Kono[3], Curtis Kenny-Benson[3], and Guoyin Shen[3]**

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A Paris-Edinburgh Press (PEP) has been commissioned at the GSECARS beamline 13-ID-C, with a Si (311) high-energy monochromator and a multi-channel collimator (MC) assembly, which consists of two arrays of fine slits (WC blades) arranged in two concentric circular arcs. Both arrays consist of 75 slits with 0.8° separation and are located 50 and 200 mm, respectively, from the center. Slit widths of the inner and outer arrays are 0.05 and 0.20 mm, respectively. By oscillating the silts during data collection, background scattering can be effectively removed. Similar MC assemblies have been used extensively with PEP at ESRF for studying metallic liquids and low-Z materials [1]. The PEP is mounted on a general purpose diffractometer [2], with an area detector (MAR CCD) mounted on the two-theta arm. With unfocused incident monochromatic beam (65 keV) collimated at 0.2 mm, 30 min is sufficient to collect weak signals of a 2 mm diameter amorphous silicate sample, with minimal background due to the surrounding solid pressure medium. An analysis shows that with a fine incident beam of 50 µm, a collimation depth of 0.5 mm can be achieved at $2\theta$ angles above 10°. To increase pressure and temperature range, we have developed a cupped-toroidal Drickamer (CTD) anvil [3]. The anvil design, with a central depression, a toroidal groove and a small tapered angle, combines features of modified Drickamer anvil and the traditional PE anvil. By optimizing the parameters for the CTD anvil design, pressures corresponding to the mantle transition zone can be generated. Cell assemblies with thermally insulating materials have been developed and temperatures up to 2000°C have been maintained steadily over hours. In the future, the large horizontally focusing Kirkpatrick-Baez mirror will be applied to focus the incident beam, thus allowing samples with diameters below 0.5 mm in diameter to be studied. The excellent spatial selectivity provides an exciting opportunity for liquid structure studies in the PEP.

*We thank Mohamed Mezouar of ESRF for the design of the MC and advice during the setup.*

[1] Morard, G., et al., *Rev. Sci. Instrum.* **82**, 023904 (2011).

[2] Rivers, M.L., et al., Properties of Earth and Planetary Materials at High Pressure and Temperature, 79–8 (1998).

[3] Kono, Y., et al., *Phys. Earth Planet. Int.* **228**, 269–280 (2014).

# Instrumentation

A-25

## Checking Intensity Corrections on a CCD Detector with a Fiber-optic Taper

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CCD detectors employing phosphors and fiber-optic tapers have been in use for more than 20 years. Thousands of structures have been solved and continue to be solved to this day. However, there is a lot of leeway in structure solution going from intensity to structure factor. Detector manufacturers attempt to account for any non-uniformity in the phosphor and light losses in fiber-optic tapers by measuring the response of the assembly to an isotropic flood field of x-rays, usually using some kind of fluorescence radiation. In this study, the same Si (3 1 1) reflection was imaged and integrated at various fiber-optic taper positions on an Area Detector Systems Corporation Quantum 210r CCD detector. Results indicate that corrections made for intensity losses in the fiber-optic taper/phosphor assembly are not adequately accounted for by the current flat-field correction, with uncorrected losses in excess of 30% near the corners of the fiber-optic taper. These results will also show that by using a simple radial fall-off correction term in addition to the flat-field correction, losses not accounted for by the current flat-field correction can be limited to less than 5%. All work was performed at beamline 19BM, at the Structural Biology Center located at the Advanced Photon Source.

*This work was supported by the U.S. Department of Energy, Office of Biological and Environmental Research, under contract DE-AC02-06CH11357.*

A-26

## FASPAX: A Fast, Integrating Detector for the APS-upgrade

**Robert Bradford[1], Davide Braga[2], Gregory Deptuch[2], Farah Fahim[2], Dave Kline[1], Tim Madden[1], and Tom Zimmerman[2]**

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FASPAX (Fermi-Argonne Semiconducting Pixel Array X-ray detector) has been planned as a fast integrating area detector with wide dynamic range for time resolved applications at the upgraded APS. The detector will achieve a burst image rate of 13 MHz, matching the bunch rate of the proposed storage ring timing mode. A unique integration circuit will permit wide dynamic range — from single photon sensitivity to $10^5$ photons/pixel in a single exposure. Incorporation of a novel interposer layer in the hybrid stack will permit large area sensors without the usual coverage gaps associated with hybrid pixel devices. The detector will provide access to the full temporal resolution of the APS, and enable novel science, such as pump-probe studies of irreversible systems.

The APS Upgrade is currently funding development of small prototypes that will be tested in the next few years. This poster will present an overview of the detector, and give some details about the ongoing development of the major detector systems, including the sensor and readout chip, mechanical design, back end readout electronics, and software.

## A-27
## Elemental Sensitivity near the Atomic Scale

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Variants of the scanning probe microscope have proven tremendously valuable for extracting detailed information about the nature of a sample's surface (atomic, electronic, magnetic); however, it has proven difficult to yield direct chemical information utilizing scanning probe techniques alone. At Argonne National Laboratory's Advanced Photon Source, a new *in situ* high-resolution microscopy technique, the synchrotron x-ray scanning tunneling microscope (SXSTM), combines synchrotron x-rays as a chemical probe and the nanofabricated metal-insulator-metal tips of a tunneling microscope as a detector. The SXSTM technique takes full advantage of 1) the chemical, electronic, magnetic and structural sensitivities that synchrotron x-ray radiation offers and 2) the sub-nanometer spatial resolution offered by the scanning tunneling microscope.

Utilizing SXSTM, chemical fingerprinting of individual nickel clusters on the Cu(111) surface has been achieved with a 2 nm lateral resolution and a chemical sensitivity confined to the first atomic surface layer. The surface sensitivity demonstrated by the SXSTM technique will enable exciting new areas of opportunity and discovery in the chemical and materials sciences.

*This work was funded by the Office of Science Early Career Research Program through the Division of Scientific User Facilities, Office of Basic Energy Sciences of the U.S. Department of Energy through Grant SC70705. Work at the Advanced Photon Source, the Center for Nanoscale Materials, and the Electron Microscopy Center was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under contract DE-AC02-06CH11357.*

[1] http://www.aps.anl.gov/Xray_Science_Division/Sxspm/.

[2] U.S. Patent Application 13/791,157.

## A-28
## Minimizing Thermal Drift in a Dual Crystal Monochromator

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Undulators on 3$^{rd}$ generation synchrotron sources such as the APS can produce high heat loads on downstream optics. User requests for different x-ray energy (undulator gap) can significantly change the power loading and induce thermal drifts in optics. A channel-cut monochromator (CCM) tends to be less sensitive to the varying thermal load, but all downstream components must track the vertical beam movement as a function of energy. A double crystal monochromator (DCM) offers an operational advantage over the CCM due to the ability to maintain constant exit-height over a wide range of energies. However, the DCM is more sensitive to varying power loads which may result in thermal fluctuations that distort the mechanics and cause intensity loss and beam position drift. Here we report a thermally stabilized DCM that incorporates PID control of the temperature of key points in the DCM. The DCM exhibits negligible thermally induced drift as the undulator gap varies from fully open to fully closed and the power load on the 1$^{st}$ crystal varies from less than 100 W to in excess of 1000 W, respectively.

*GM/CA@APS has been funded in whole or in part with Federal funds from the National Cancer Institute (ACB-12002) and the National Institute of General Medical Sciences (AGM-12006). This research used resources of the Advanced Photon Source, a U.S. Department of Energy (DOE) Office of Science User Facility operated for the DOE Office of Science by Argonne National Laboratory under Contract No. DE-AC02-06CH11357.*

## A-29

## Spherically Bent Quartz Analyzer Characterization at 1-BM

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The bending magnet beam line 1-BM at the Advanced Photon Source was reconfigured recently, in part, as a test bed for hard x-ray optics. The beamline has a Si(111) × Si(111) double crystal monochromator and covers the energy range from 6 to 28 keV. The beam size available is 100 mm horizontally and 5 mm vertically. This paper reports on a study of a spherically bent crystal analyzer typical of those used for plasma spectroscopy or x-ray imaging. The crystal is alpha-quartz, 60 mm by 40 mm, which is bent by optical (direct) contact to a glass substrate that has been polished and ground to a 672 mm radius of curvature. The crystal had been ground and polished down to 0.1 mm thickness with the surface parallel to the (1 0 -1 1) crystal plane. Bragg case rocking curves and x-ray topographs were obtained at 12.75 keV for the (3 0 -3 3) reflection at a Bragg angle of 25.86 deg. A rocking curve with a very small vertical beam was relatively narrow, but a rocking curve obtained with a large vertical beam was very wide corresponding to the range of spherical curvature sensed in the vertical diffraction plane. For this large vertical beam , a continuous-exposure topograph obtained by rocking slowly across the full rocking curve with the same film in place showed a surprising amount of structure and revealed a strain pattern corresponding to several round features of roughly 0.3 mm diameter. These features are interpreted presently as due to gas bubbles formed and trapped at the interface between the bent crystal wafer and the spherical form during the bonding process.

*We acknowledge E.O. Baronova for the fabrication of the analyzer. The beamline development work at the APS was supported by the U.S. Department of Energy, Office of Science, under Contract No. DE-AC-02-06CH11357.*

## A-30

## Failure Modes of OFE and GlidCop® Copper Absorber Materials under X-ray Induced High Heat Load Thermal Fatigue Conditions

**Gary Navrotski, Mike Bosek, Jeff T. Collins, Jeremy Nudell, Ali Khounsary, and Patric Den Hartog**

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Water cooled, high thermal conductivity, oxygen free copper and dispersion strengthened copper composites (GlidCop®) are workhorse absorber materials as strike surfaces for synchrotron x-ray masks, shutters and stops around the world. Although conservative design criteria keep these materials from ever approaching failure, a knowledge of the material's failure modes allows for more informed discussions of the safety factors inherent in using established design limits.

Thermomechanical fatigue tests were conducted under normal incidence x-ray thermal loading from low power, up to limit power levels of 4,800 W at peak power density of 430 W/mm$^2$. The materials respond to these increasing power loads in a progressive and logical fashion. In oxygen free copper (UNS C10100) increased thermal loading induces surface twinning, recrystallization, grain pop-out and surface rumpling leading to a roughened strike surface. In dispersion strengthened copper (UNS C15715) failure modes progress through stages from surface fiber drop-out or "cat scratching," crack generation, surface heaving, crack growth, surface folding and eventual melting.

This poster presents metallurgical and microstructural, observations and measurements on these materials over the scope of thermal loading conditions that can be tolerated in synchrotron x-ray components.

## A-31
## Vibrations, Spot Size, and Flux Measurements at XSD Beamlines

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The proposed Multi-Bend Achromat lattice replacing the existing Double Bend lattice at the APS will reduce the beam emittance by two orders of magnitude. Several new beamlines are being considered to take full advantage of the brightness increase and others should be upgraded to increase their capabilities.

To this end, we have started measuring the performance of the XSD beamlines to identify issues that could limit their performance in the new lattice. We have measured the spot size, flux, and vibrations of several beamlines.

The results of the measurements are interpreted using ray tracings and wave propagations using the SHADOW1 and the Hybrid2 code. In general, the measured flux and the calculated flux are in good agreement. The measured source sizes are usually larger than the calculated ones, but can be explained by assuming larger figure errors than those measured over 10 years ago. We have identified several vibration frequencies common in several beamlines, some of which are present in the electron beam.

*The Advanced Photon Source, an Office of Science User Facility operated for the U.S. Department of Energy (DOE) Office of Science by Argonne National Laboratory, was supported by the U.S. DOE under Contract No. DE-AC02-06CH11357.*

[1] Sanchez del Rio, M., Canestrari, N., Jiang, F., and Cerrina, F. (2011). *J. Synchrotron Rad.* **18**, 708–716.

[2] Shi, X., Reininger, R., Sanchez Del Rio, M., and Assoufid, L., *J. Synchrotron Rad.* **21**, 669–678 (2014).

## A-32
## Vortex® SDD X-ray Spectrometers with Improved Counting Rate Performance

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Silicon Drift Detectors (SDD) are most often used the Energy Dispersive Spectrometer (EDS) for x-ray microanalysis, x-ray fluorescence (XRF), total reflection XRF (TXRF), XRF imaging and x-ray absorption spectroscopy (XAS). Also the SDD spectrometers are very popular in synchrotron based experiments such as XAFS (x-ray absorption fine-structure) and XANES (x-ray absorption near-edge structure) because of their high-count rate capability and high energy resolution.

We will present several aspects of our design efforts toward the development of the SDD XRF spectrometers with an extremely high counting rate performance. Our spectrometers are based on the Vortex® SDD with the sensitive area of 50 $mm^2$ and the thickness of 0.5 mm and 1 mm. A new advanced front-end ASIC preamplifier integrated with the Vortex® SDD enables to increase the output count rate up to 850 Kcps at about 50% DT with the energy resolution (FWHM at 5.9 keV) better than 250 eV when it is used with conventional pulse processing electronics. With the latest adaptive pulse processing electronics achievable output count rate is even higher than 1 Mcps at relatively low DT and with the energy resolution better than 200 eV. However, despite the high count rate capability of the Vortex® SDD, the performance of the spectrometer is limited by a signal processing electronics. Therefore, to get a further increase in count rate capability we have developed multi-element SDD spectrometers with several design configurations including options for ultra high vacuum applications. A detailed data concerning the total solid angle of different Vortex multi-element detectors, their energy resolution and output count rate performance will be presented.

## A-33

## Low Temperature Synchrotron X-ray Scanning Tunneling Microscopy (LT-SXSTM)

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Low temperature scanning tunneling microscopy (LT-STM) combined with synchrotron based x-rays provides a new tool to capture chemical interactions and magnetic spin states on surfaces at high spatial resolution. The technique will drastically increase the spatial resolution, and it measures chemical and magnetic information along with surface topography. Here, we will present the current status of the ongoing development of a LT-SXSTM. The system is equipped with, high stability 4 axis stages, a half focusing polycapillary and positioners, a vacuum suitcase for sample and tip exchanges, custom designed STM head and a liquid Helium flow cryostat.

## A-34

## Mechanical Design of Multiple Fresnel Zone Plates Precision Alignment Apparatus for Hard X-ray Focusing in Twenty-nanometer Scale

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To overcome the limitations in today's fabrication techniques for high-efficiency Fresnel zone plates (FZPs) capable for hard x-ray focusing in the twenty-nanometer scale, a new approach of stacking FZPs in an intermediate-field was published by Vila-Comamala et al. in 2012 [1]. With this approach, a precision alignment apparatus for multiple FZPs handling and aligning must be designed to meet the following challenging design requirements: 1) Each of the stacking FZPs need to be manipulated in three dimensions with nanometer-scale resolution and travel range of several millimeters. 2) The relative three-dimensional stabilities between all of the stacking FZPs (especially in the x-ray beam transverse plane) are required to be kept within few nanometers for more than eight hours, the duration of the hard x-ray focusing for nanoprobe operation. 3) Compatible with the operation of multiple optics configurations, such as switchable optics between FZPs and K-B mirrors, for the APS future x-ray nanoprobe design.

Several prototypes have been designed and tested at the APS [2,3]. In this poster we present the precision mechanical design of the apparatus prototypes for two, three, and six FZPs alignment in an intermediate-field, as well as the test results of their hard x-ray focusing performances.

*Work supported by the U.S. Department of Energy, Office of Science, under Contract No. DE-AC02-06CH11357.*

[1] J. Vila-Comamala, M. Wojcik, A. Diaz, M. Guizar-Sicairos, C.M. Kewish, S. Wang, and C. David, (2012). *J. Synchrotron Rad.* **20**, 397–404.

[2] S. Gleber, M. Wojcik, J. Liu, C. Roehrig, M. Cummings, J. Vila-Comamala, K. Li, B. Lai, D. Shu, and S. Vogt, *Optics Express* Vol. **22**, Issue **23**, DOI: 10.1364/OE.22.028142, 2014.

[3] D. Shu, J. Liu, S.C. Gleber, B. Lai, J. Maser, C. Roehrig, M.J. Wojcik, and S. Vogt, U.S. Patent application in progress for ANL-IN-13-092.

## A-35
## Rocking Curve Imaging at 1-BM X-ray Optics Test Beamline

**Stanislav Stoupin, Yuri Shvyd'ko, Emil Trakhtenberg, Zunping Liu, Keenan Lang, Xianrong Huang, Michael Wieczorek, Elina Kasman, Lahsen Assoufid, and Albert Macrander**

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We report progress on implementation and commissioning of rocking curve imaging capability [1] at 1-BM Optics Test Beamline of the Advanced Photon Source. Si collimator crystals of various crystallographic orientations were designed and fabricated using in-house capabilities to accommodate growing needs of strain characterization in diffractive crystal optics and other semiconductor single crystals (e.g., crystal wafers). Quality of the Si collimator crystals was characterized using monochromatic beam topography across the entire working crystal surfaces. The initial design features evaluation of strain in single crystals in the nearly-nondispersive Bragg double-crystal geometry. Preliminary monochromatization of the x-rays is performed using a double-crystal Si 111 monochromator (DCM). An area detector (CCD) imaging the double-reflected beam permits sequential acquisition of x-ray topographs at different angular positions on the rocking curve of the crystal under investigation. Results on sensitivity and spatial resolution in imaging crystal strain are presented. The new setup complements laboratory-based x-ray topography capabilities [2–4] of the Optics group at the Advanced Photon Source.

[1] Lubbert, D., Baumbach, T., Hartwig, J., Boller, E., and Pernot, E., *Nucl. Instrum. Methods Phys. Res. Sect. B* **160**, 521–527, (2000).

[2] S. Krasnicki, *Rev. Sci. Instrum.* **67**, 3369 (1996).

[3] A.T. Macrander, S. Krasnicki, Y. Zhong, J. Maj, and Y.S. Chu, *Appl. Phys. Lett.* **87** (19), 194113 (2005).

[4] S. Stoupin et al., *J. Appl. Cryst.* **47**, 1329, (2014).

## A-36
## The APS Detector Pool

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The APS Detector Pool provides users with access to many different types of x-ray detectors and related equipment. The most popular devices include: Area Detectors (Pilatus 100K, PixiRad, Mar 165 CCD), Microscopy Cameras (Andor Neo, Prosilica), Spectroscopic Detectors (Vortex ME4, Oxford Ge, Amptek CZT), Point Detectors (Oxford Cyberstar, Mythen silicon strip, PIN Diodes), and Temperature Control Stages (Linkam, CryoStream). It also coordinates equipment loans between beamlines, provides hardware and EPICS troubleshooting, and works with vendors to circulate demo units of next generation detectors.

## A-37
## New Mini-beam Collimator Provides Lower Background

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The GM/CA-developed, quad-mini-beam collimator, and advanced rastering and vector data-collection software tools, have enabled successful data collection on some of the most challenging problems in structural biology. This is especially true for membrane-protein crystals grown in lipidic cubic phase, where crystals are typically small, fragile, and "invisible" when cryo-cooled.

The current quad mini-beam collimators have 5-, 10-, and 20-μm beam-defining apertures with 250 μm exit apertures, and a 300 μm aperture (ScatterGuard) for the full beam with a 600 μm exit aperture. Due to tolerances in the alignment of each beam defining aperture/exit aperture pair, two motorized translations and two manual angle adjustments are required for optimal alignment.

Tests with a new prototype mini-beam collimator with 5 μm beam defining aperture and a 150 μm exit aperture in combination with a 0.5 mm diameter beam stop have shown that the background can be further reduced. The design and test results of the new and upgraded mini-beam collimator will be presented.

### A-38
### Rapid *in situ* X-ray Position Stabilization via Extremum Seeking Feedback

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The Double Crystal Monochromator (DCM) 2nd crystal is one of the primary sources of intensity and position instabilities. While efforts to provide the 2nd crystal with thermal stabilization, vibration isolation, and strain relieved cables have substantially attenuated externally driven instabilities, long term drift remains a tedious problem requiring occasional intervention. Here, we address this problem using simultaneous Extremum Seeking Feedback Control (ESFC) for monochromator x-ray flux and *in situ* vertical beam position stabilization. Monochromator flux intensity stabilization is achieved using previously demonstrated extremum seeking feedback [1–5]. Intensity gradient detection for position ESFC is achieved by exploiting pre-existing vertical oscillations used for monochromator flux stabilization. Flux recovery at the sample after a 2 keV energy move is < 6 seconds and intensity stability through a 5 μm aperture is 1.5% FWHM over a period of 8 hours.

[1] R. Fischetti, S. Stepanov, G. Rosenbaum, R. Barrea, E. Black, D. Gore, R. Heurich, E. Kondrashkina, A. Kropf, S. Wang, K. Zhang, T. Irving, and G. Bunker, "The BioCAT undulator beamline 18ID: a facility for biological non-crystalline diffraction and x-ray absorption spectroscopy at the Advanced Photon Source," *Journal of Synchrotron Radiation* **11**, p. 399, 2004.

[2] N. Igarashi, K. Ikuta, T. Miyoshi, N. Matsugaki, Y. Yamada, M. Yousef, and S. Wakatsuki, "X-ray beam stabilization at BL-17A, the protein microcrystallography beamline of the Photon Factory," *Journal of Synchrotron Radiation* **15**, p. 292, 2007.

[3] D. Mills and V. Pollock, "Stabilizing feedback system for synchrotron radiation monochromators," *Review of Scientific Instruments* **51**, p. 1664, 1980.

[4] S. Stoupin, F. Lenkszus, R. Laird, K. Goetze, K. Kim, and Y. Shvyd'ko, "Nanoradian angular stabilization of x-ray optical components," *Review of Scientific Instruments* **81**, p. 055108, 2010.

[5] R. van Silfhout, A. Kachatkou, E. Groppo, C. Lamberti, and W. Bras, "Position and flux stabilization of x-ray beams produced by double-crystal monochromators forEXAFS scans at the titaniumK-edge," *Journal of Synchrotron Radiation* **21**, p. 401, 2014.

## Materials Science

### A-39
### ALD-grown Pd Nanoparticles Supported on $TiO_2$- and SrO- terminated $SrTiO_3$ Nanocuboids

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We report the application of $SrTiO_3$ (STO) nanocuboids with well-defined {001} facets as the support for Pd nanoparticles deposited by atomic layer deposition (ALD). The advantage of using a support with well-defined surfaces is that the surface resembles a model catalyst but retains a high surface area enabling catalytic reaction studies. This approach bridges the materials gap between single crystal models and catalysts with more complex structures. We demostrate how the surface termination ($TiO_2$- or SrO) influences the growth mode, morphology and the chemical properties of the supported Pd nanoparticles. The morphology and chemical nature of the Pd particles were studied by transmission electron microscopy (TEM), x-ray scattering, and x-ray absorption fine structure

(XAFS) measurements. We also report that the effective coverage, chemical state, and the size of Pd nanoparticles can be controlled by the number of ALD cycles. CO oxidation reaction was used as a probe to determine and compare the catalytic performance of the Pd nanoparticles supported on $TiO_2$- or SrO- terminated STO nanocuboids. The study helps demonstrate how to prepare supported catalysts on mixed metal oxide supports.

## A-40
## Understanding the Complex Structure of Composite, Lithium-ion Cathode Materials

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Composite electrode materials, as indicated in the notation $y[xLi_2MnO_3•(1\text{-}x)LiMO_2]•(1\text{-}y)LiM_2O_4$ (M=Mn,Ni,Co), are ideally comprised of several structural motifs. One is the prototypical, layered *R-3m* ($LiMO_2$) structure, one is that of the layered, monoclinic (*C2/m*) $Li_2MnO_3$, and the third is the spinel (*Fd3m*) $LiM_2O_4$. Such compositions have shown promising electrochemical performance as lithium-ion cathode materials and are currently being studied as such. In order to fully ascertain the actual promise of these materials a better understanding of their structural and elemental compositions is necessary. However, the nanoscale integration of the different components, along with local, chemical inhomogeneity, gives rise to extremely complex overall structures. Furthermore, once cycled in real cells, these structures change in ways that are not yet understood.

This project focuses on advanced characterization of pristine, end-member and composite structures in order to build a "baseline" understanding that can be used for future studies on working, composite electrode structures of all types.

## A-41
## Ferroelectric Domain Engineered Strain Investigated Using Diffuse Multiple Scattering

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The physical properties and functional efficiency in many real systems are intimately related to their internal stresses and strains. Thus, it is possible to control and tailor the functional properties of a material through modification of its structure. Different effects are observed in bulk crystals, polycrystalline thin films or in epitaxial nanostructures due to the differences in the dimensionality of any long-range order. Recently, there has been considerable research effort on understanding the complex interplay between sample structure and the internal strain which gives rise to the ferroic orders that can be observed in a wide range of materials including the important $(1-x)\,[Pb(Mg_{1/3}Nb_{2/3})O_3]-x\,[PbTiO_3]$ (PMN–PT or $PbZn_{(1-x)}Ti_xO_3$ (PZT). Of particular technological relevance is the correlation between strain and electric polarisation in these systems which we are exploiting, through the European Metrology Research Programme's Nanostrain project, to develop a new Piezoelectric-Effect-Transistor (PET). Suitably patterned architectures of these materials may offer a new route to replace aging CMOS technology with increased speed (x10) and, critically, significantly lower power consumption (~x100 less energy). To address this transformative technology the Nanostrain project brings together several European national laboratories in a consortium including nine commercial companies.

In this poster, we describe the first application of a new diffraction metrology similar to Rutherford, Kikuchi and Kossel lines and named Diffuse Multiple Scattering lines (DMS) [1]. As the DMS features arise from the crystal truncation rods they act as divergent secondary sources within the sample and hence have the crystallographic information encoded. Following a Laué description, the DMS appear as cones/circles on a 2D detector [1]. The newly developed experimental metrology, supported by robust modelling has several noteworthy implications: 1) the strain (and its dispersion) can be determined without moving the sample and in a single exposure. This allows a much wider range of sample environments (*in situ* application of electric/magnetic fields, temperature, etc.) to be used with significantly reduced errors than more conventional strain measurements, 2) where two, or more, features intersect the lattice parameter determination is very precise [2], 3) unlike Kossel lines [3,4], the sampling volume can be tuned through the x-ray energy. We report the application of DMS to domain engineered single crystal PMN-PT samples whilst under the application of an external electric field. We will show how DMS can extract the subtle evolution in both the lattice parameters and the strain state with E-field in real time. These measurements form a critical step in exploiting ferroelectric materials in general, and are crucial in our continuing efforts to develop a PET device.

[1] Nisbet et al., *Acta Cryst.* **A71**, 20–29 (2015).

[2] Lonsdale et al., *Adv. Mater.* **21**, 817–823 (2009).

[3] Robach et al., *J. Appl. Cryst.* **44**, 688–696 (2011).

[4] Däbritz et al., *Appl. Surf. Sci.* **179**, 38–44 (2001).

## A-42

## Probing the Role of Ga in Amorphous Conducting Oxides through Local Structure Studies

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The study of amorphous (a-) conducting oxides is an emerging field. The lack of grain boundaries, smooth surfaces, and low temperature deposition position these materials as ideal candidates for large area applications and flexible electronics. Most impressively, these materials maintain high electron mobility in the amorphous state. These benefits have led the recent commercialization of a-IGZO (Ga and Zn doped indium oxide) as a replacement for a-Si as the channel layer of thin film transistors in display technology. Despite this success, fundamental understanding of structure-property relationships is still lacking and must be improved to guide further development of amorphous conducting oxides. X-ray absorption spectroscopy (XAS) is one of the few tools that can be used to probe the structure of amorphous materials. Amorphous indium oxide doped with Ga (a-IGO) is a model system to help develop the role of dopants in amorphous oxides. An in depth XAS study was carried out to determine inter-atomic distances, coordination numbers, and structural disorder parameters as a function of Ga doping level. The correlation between XAS-derived structural features and the dopant-dependent evolution of both electrical properties and thermal stability of a-IGO will be discussed.

*This work is supported by the NSF MRSEC Program No. DMR1121262.*

## A-43
## High-energy X-ray Techniques for Studying Nuclear Energy Relevant Materials

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High performance materials that can withstand intense radiation fields, elevated temperatures, multi-axial stresses, and corrosive environment and nuclear fuel technologies that minimize waste and fully-utilize the fuel's stored energy are necessary for the deployment of advanced nuclear energy systems and the expansion of nuclear energy as a reliable, affordable, and clean energy source.

High energy x-ray techniques at the Advanced Photon Source (APS) are a unique set of nondestructive tools that can be used to understand the structure-processing-property relationship, characterize the thermos-mechanical state, and investigate failure mechanisms in polycrystalline nuclear materials *in situ*. In this poster, we illustrate the use of these high energy x-ray techniques to better understand polycrystalline materials employed in nuclear engineering applications.

## A-44
## *In situ* XAS Investigation of an Iron Oxide Battery Anode in Aqueous Electrolytes: Effect of Surface Modification on Rheology and Electrochemistry

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Iron oxides have been demonstrated to be anode materials in aqueous environments and multiple literature reports [1] exist proposing possible redox events that can occur in the system such as Fe(3+)/Fe(2+), Fe2+/Fe(0) and accompanying lithiation. However, the nature of the redox event varies with several factors such as repeated electrochemical cycling, nature of electrolyte used, concentration of electrolyte used and rate of charge/discharge. In order to optimize the system to a certain capacity and operational conditions, it is important to understand the nature of the redox transition occurring during electrochemical cycling and how to control it. Furthermore, performance in aqueous environments have been found to be heavily dependent on the lithium ion content of the electrolytes; [1] higher concentrations facilitating the reduction process and improving columbic efficiency. However, a satisfactory explanation currently does not exist and further analysis is required to fully establish the nature of redox events.

This study reports on the use of XAS as an investigative tool to study the reversible redox chemistry of Iron (III) oxide nanoparticles, establish the role of Li+ and correlate redox events with coulombic input. SEM and XRD data in conjunction with *in situ* XAS analysis reveals that the Iron oxide nanoparticles can be fully reduced to metal in aq. 5M LiOH electrolytes, but leads to growth of large micron sized crystals. A simple surface modification procedure has also been developed for the nanoparticles to facilitate preparation of low viscosity nanofluids and prevent growth of crystallites due to repeated electrochemical cycling as observed in the pristine material. SEM, XRD and *in situ* XAS data are collectively used to understand these phenomena in both pristine and modified nanoparticles. This study forms the basis for electrochemical and rheological testing of the same system as a suspension type nanoelectrofuel electrode. [2]

[1] U. Casellato, N. Comisso, and G. Mengoli, "Effect of Li ions on reduction of Fe oxides in aqueous alkaline medium," *Electrochimica Acta* **51** (2006) 5669–5681

[2] E.V. Timofeeva, J.P. Katsoudas, C.U. Segre, and D. Singh, "Rechargeable Nanofluid Electrodes for High Energy Density Flow Battery," *Cleantech 2013,* Chapter 9, Energy storage, pp. 363–366.

## A-45

# Micro CT Microscopy and Ultra Small-angle Neutron Scattering Characterization of Alkali-Silica Reaction (ASR) Gel Development in Mortar Specimens with Recycled Glass Particles

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The alkali-silica reaction (ASR) (or alkali-aggregate reaction) is a major distress in cementitious concrete and structures due to the expansion damage initiated from reactive siliceous aggregates. The ASR takes place between the reactive silica contained in aggregates and the alkalis in pore solutions of cement paste. The alkali-silica reaction is initiated by a reaction between the hydroxyl ions in the pore solution and certain types of silica in the aggregate. Once the ASR gel is formed, the gel will expand by imbibing water. The driving force for expansion and cracking is the osmotically-derived imbibition of water into the insoluble reaction gel. This study aims to characterize the microstructure of alkali-silica reaction gels formed from alkali solutions and to capture the ASR damage development with different reaction conditions by utilizing the X-ray Micro-tomography and Ultra Small Angle Neutron Scattering (USANS).

To better understand this process, an accelerated chemical reaction between glass particles and strong alkali solution was conducted to simulate the ASR reaction in reality. The cement mortar samples were prepared using recycled glass particles (mesh size 20–40) as fine aggregates with a volume percentage of 30% and a water to cement ratio (w/c) of 0.5. Cement mortar samples were molded in plastic tubes with an inner diameter of 2 mm. After hardening for 24 hours, the samples were merged into different concentration NaOH solution for curing. The ASR gel (with lower density) and microcracks can be identified from the x-ray CT images. The damage development in glass particles were also observed. To further investigate the microstructure of ASR gel, the 3D porous gel microstructure was reconstructed and the average averaged size of ASR gel particle was determined as 2.88~2.92 μm. With USANS data, the microstructural properties such as surface area and porosity at micron scales were obtained by analyzing the I-Q data with the Porod scattering theory [1]. An Guinier-Porod model [2,3] was used to determine the size and dimensionality of the reacted Alkali-silica gel scattering data. The analyzed ASR gel size dimensions from USANS match well with CT image data.

[1] Porod, G. The x-ray small-angle scattering of close-packed colloid systems. Kolloid Z, 1951. **124**: p. 83.

[2] Guinier, A., G. Fournet, C.B. Walker, and K.L. Yudowitch. Small-angle scattering of x-rays. 1955.

[3] Glatter, O. and O. Kratky, Small angle x-ray scattering. 1982: Academic press.

## A-46

# Atomic Layer Deposition of Doped Films Studied by *in situ* Synchrotron Techniques with a Mobile and Modular Reactor

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Progress for understanding growth of thin films by atomic layer deposition (ALD) has been aided by application of synchrotron studies over the past ten years. ALD is a vapor phase deposition technique, similar to chemical vapor deposition, where precursors are introduce subsequently, in two complementary half-cycles, to grow thin films. In ALD, film growth is controlled by chemically self-limiting reactions at the gas/surface interface, which produces conformal films blind to aspect ratio of trenches and intricate nano-structures. While significant advancements have been made by other *in situ* techniques, such as quartz-crystal microbalance, infrared spectroscopy and ellipsometry, the power of *in situ* synchrotron studies will provide wealth unique information. To take advantage of this an ALD apparatus was designed to integrate with the APS.

The ALD apparatus consists of common process hardware (precursor delivery, temperature and pressure control, and heater supply) and a set of reactor chambers which are specifically tailored for compatibility with individual x-ray techniques (or groups of techniques) and incorporated into the portable ALD system in a plug-and-play fashion. X-ray scattering studies are enabled by an open-top reactor cell which can be fitted with either a hemispherical graphite dome or a kapton/mica/sapphire window assembly. X-ray absorption spectroscopy (XAS) is performed on flat substrates in a grazing incidence mode using a closed reactor cell with removable kapton windows. Both reactor cells are compatible with a standard Huber threaded goniometer mount.

To demonstrate the capabilities of the *in situ* ALD tool, initial results from three separate experiments will be presented. 1) *In situ* crystal truncation rod (CTR) and reflectivity (XRR) measurements of epitaxial ALD growth of ZnO on c-plane sapphire at 150°C were performed at 33-BM-C. 2) *In situ* grazing incidence small angle x-ray scattering (GISAXS) at 12-ID-B was employed to study island nucleation and coalescence of ALD MnO and ZnO at 150°C. 3) The coordination environment of low dopant levels of $Er_2O_3$ and the half-cycle ALD chemistry of erbium incorporation into amorphous $Al_2O_3$ and crystalline MgO, both grown by ALD on c-sapphire, were examined at 300°C with *in situ* XANES/XAFS at 20-ID-B.

*Argonne National Laboratory's work was supported by the U.S. Department of Energy, Office of Science under Contract No. DE-AC02-06CH11357.*

## A-47
## A Novel Phase in Li-Si System

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Li15Si4, the only crystalline phase that forms during lithiation of the Si anode in lithium-ion batteries, was found to undergo a structural transition to a new phase at approximately 7 GPa. Despite the large unit cell of Li15Si4 (152 atoms in the unit cell), *ab initio* evolutionary metadynamics (using the USPEX code) successfully predicted the atomic structure of this new phase (beta-Li15Si4), which has an orthorhombic structure with a Fdd2 space group. In the new beta-Li15Si4 phase the atomic packing is more efficient owing to the higher Si-Li coordination number and shorter Si-Li, Li-Li bonds. Beta-Li15Si4 has substantially larger elastic moduli compared with alpha-Li15Si4, and has good electrical conductivity. As a result, beta-Li15Si4 has superior resistance to deformation and fracture under stress. The theoretical volume expansion of Si would decrease 25% if it transformed to beta-Li15Si4, instead of alpha-Li15Si4, during lithiation. Moreover, beta-Li15Si4 can be recovered back to ambient pressure, providing opportunities to further investigate its properties and potential applications.

In addition, bulk modulus of $Li_{15}Si_4$ was obtained experimentally for the first time (B=28.4±0.6 GPa), and shear modulus and Poisson's ratio were calculated as well. We found that the bulk modulus of $Li_{15}Si_4$ follows a linear interpolation relationship between the bulk moduli for pure Li and Si. These results provide valuable experimental data to validate the theoretical calculation and are also important inputs for modeling the Li-Si system.

# Nanoscience and Nanotechnology

A-48

## Metrology of Positioning Systems at the Nanometer Level

**Roger Burg and Byron Fruit**

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Verifying claims of performance or even simply characterizing performance in a positioning system at the sub-micrometer to nm-level is a non-trivial problem. To do this requires specific equipment and software as well as specialized knowledge about the tools, techniques and best practices. In this paper, we show performance data obtained on various stages and how both the system-design and metrology methods affect the quality of the results.

A-49

## Enhancing Solar Cell Performance through the Use of Plasmon Generating Nanocomposite Materials

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Over the last ten years Mantis Deposition has developed an ultra-high vacuum compatible nanoparticle source capable of producing a wide range of nanoparticle structures with exquisite control over their composition and size. In collaboration with our customers we have demonstrated the capability of this technology in several sectors including energy, surface science, catalysis, biotechnology, and information technology. In this presentation we will describe how the properties of particles on the nanometer scale are being harnessed to enhance the performance of solar cells. We will show how plasmon generating nanocomposite materials can be tuned to optimise the absorption wavelength by controlling the diameter of silver nanoparticles in various thin film structures. We will also outline some of the outstanding challenges and potential strategies on the road to exploitation of this technique.

A-50

## *Operando* Investigation of the Hydriding Phase Transformation in Single Palladium Nanocubes

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Phase transitions in reactive environments are crucially important in energy and information storage, catalysis, and sensors. Nanostructuring materials used in these systems can cause a host of desirable properties, including faster charging/discharging kinetics, increased lifespan, and record activities. However, establishing the causal link between structure and function is challenging for nanoparticles as ensemble measurements convolve intrinsic single particle properties with sample size and shape diversity. Here we study the hydriding phase transformation in individual palladium nanocubes under operando conditions using coherent x-ray diffractive imaging. We directly observe two-phase coexistence in the single particle diffraction data. The phase transformation initiates at the corner of the cube, penetrates further into the particle, and eventually violently rearranges the crystal structure. The strain distributions of the $\alpha$ and $\beta$ phases are markedly different, indicating more than a simple Wulff geometric construction is required. A phase field model is constructed to interpret the phase transformation. Our results provide a general framework for understanding phase transformations in individual nanocrystals under operating conditions in reactive environments while highlighting the utility and importance of single particle investigations to truly understand important systems.

A-51

## X-ray Absorption Spectroscopy (XAS) Study of Sorption Mechanisms of Cd(II) to Hematite ($\alpha$-$Fe_2O_3$) Nano Particles with Varying Size and pH Environment

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Natural mineral nanoparticles have gained attention due to their wide occurrence in soil and high sorption capacities for cationic and anionic contaminants influencing the mobility and transport of contaminants in the environment. This is a consequence of their small size and non-optimal surface coordination environment compared to the bulk. Hematite (alpha-$Fe_2O_3$) is a mineral whose particles are found in the nano-sized region. This study involves investigating the sorption mechanisms of the highly toxic heavy metal cadmium to hematite. Experiments were normalized to total hematite nanoparticle surface area within reaction vessels. The sorption mechanisms were investigated under different hematite particle sizes (8 nm and 40 nm surface area/mass normalized (SAN/MN)) and different pH environments (pH 7.5 and pH 9).

The adsorption edge experimental results suggested particles sorbed more Cd(II) as the pH environment was increased and the particle size was decreased. For the larger particles, the sorption edge was shifted to the right approximately by 1 pH unit. X-ray absorption near edge structure (XANES) results on 8 nm nanoparticles and the 40 nm SAN at pH 7.5 indicated the presence of similar coordination environment around the absorbing Cd atom. Cd was adsorbed to 8nm particles at pH 7.5 but did not form a precipitate whereas at pH 9, minor amounts of $CdCO_3$ and CdO were present. When experiments were normalized to hematite surface area, particle size did not substantially affect the sorption mechanism at pH 7.5. However, at pH 9 a combination of $CdCO_3$ precipitate and adsorption complex(es) were formed of which contribution from the precipitate was larger. When experiments were normalized to hematite mass, more Cd precipitation was observed in larger nano particles at both pH values. This is most likely due to the presence of fewer surface adsorption sites on the particles. Extended x-ray absorption fine structure (EXAFS) results revealed binding site details as a function of pH and particle size and will be presented.

Application of XAS technique for the purpose of gaining insights into the mechanisms of interaction between hematite nano particles and heavy metal cadmium suggests a first step towards alleviating nano particle related environmental and health hazards.

# Other

## A-52
## Globus Data Publication Services and Their Application at the Advanced Photon Source

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Globus is software-as-a-service for research data management, used at dozens of institutions and national facilities for moving, sharing, and publishing big data. Recent additions to Globus include services for robust data publication, cataloging, and discovery to support researchers with a unified set of data tools that spans the entire data lifecycle, from data capture, to active phase collaboration and sharing, to final dataset publication. These services allow users and institutions to: 1) enable publication of large research datasets with flexible policies; 2) grant the ability to publish data directly from local storage, institutional data stores, or from self-managed cloud storage, without third party publishers; 3) build extensible domain-specific metadata that describe the specific research attributes; 4) develop publication workflows and automate experiment workflows to meet institutional and researcher requirements; 5) deploy public and restricted collections that define control over who may access published data; and 6) access a rich discovery model that allows others to search, interrogate, and build upon published data.

In this poster, we will discuss Globus [1] services for data publication, cataloging, and discovery and their application at the Advanced Photon Source to high-energy diffraction microscopy, tomography, and diffuse scattering. Specifically, we will discuss how these services are being utilized to develop automated workflows that leverage high-performance compute and storage resources to stage and synthesize results from experiments and simulation; to enable near real-time visualization and inspection of complex datasets; to catalog experiment and simulation parameters, in conjunction with other dataset provenance, in an intuitively searchable form; and to present simple web interfaces for users to interact with as well as robust API and CLI to facilitate incorporation into existing data capture workflows.

[1] I. Foster. *IEEE Internet Computing* **15** (2011): 70.

## A-53
## Using Focused X-ray Pulses to Probe Microelectronic Devices for Single Event Effect Susceptibility

**David Cardoza[1], Stephen D. LaLumondiere[1], Michael A. Tockstein[1], Nathan P. Wells[1], Dale L. Brewe[2], Kevin M. Gaab[1], William T. Lotshaw[1], and Steven C. Moss[1]**

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Integrated circuits (ICs) scheduled for use in satellite electronic systems need to withstand the harsh radiation environment of space [1]. Energetic particle strikes on an integrated circuit can generate a conduction band carrier track along their trajectory that results in current or voltage transients which can disrupt the normal operation of the IC, and potentially debilitate entire electronic systems. It is of the utmost importance that all ICs under consideration or scheduled for on-orbit deployment are tested beforehand so that their susceptibilities to these single event effects (SEEs) are documented, characterized, and if possible mitigated [2].

In order to fully understand SEE susceptibilities, it is important to develop methods that can simulate the effect of charged particle strikes (i.e., a method that can deposit charge in a highly localized area around individual circuit elements such as transistors, and measure the IC's response to the impulsive charge deposition). In principle, using a beam of relativistic particles with low flux, such that a single particle would be incident on a transistor at any given time, would be the ideal method. In practice, however, no known sources allow for the regular placement of incident ions with high spatial resolution and accuracy. Lasers can be used to generate conduction band carriers and inject current into a circuit, but most wavelengths are too long to be focused to a spot size corresponding to an individual circuit element in sub-100 nm fabrication technologies. Furthermore, lasers will not penetrate metallization layers which can cover virtually all of the functional area on newer ICs. In contrast to heavy ion or pulsed laser techniques, x-rays have properties that are advantageous for characterizing individual circuit elements on microelectronic circuits — they can be focused to extremely tight spots and they can penetrate metallization. We have previously used the Advanced Photon Source to show that focused x-ray pulses can generate charge transients in microelectronic devices. These measurements were highly successful and a paper on the technique has been recently published [3]. We will present data from our recent APS measurement campaigns that compare x-ray induced transients with those produced by both lasers and heavy ions. We will show that having the capability of focusing high energy radiation provided by synchrotrons like the APS on localized spots on microelectronic circuits of high value for SEE susceptibility testing.

[1] A.L. Vampola, "The hazardous space particle environment," *IEEE Trans. Plas. Sci.,* vol. **28**, pp. 1831–1839, 2000.

[2] R.A. Reed, et al., "Single-event effects ground testing and on-orbit rate prediction methods: the past, present, and future," *IEEE Trans. Nucl. Sci.,* vol. **50**, pp. 622–634, 2003.

[3] D. Cardoza, et al., "Single Event Transients Induced by Picosecond Pulsed X-ray Absorption in III-V Heterojunction Transistors," *IEEE Trans. Nucl. Sci.,* vol. **59**, pp. 2729–2738, 2012.

## A-54

## Swift Parallel Scripting for Fast, Productive Beamline Data Analysis

**Hemant Sharma[1*], Justin Wozniak[2,3], Jun Park[1], Guy Jennings[1], Ian Foster[2,3,4], Jonathan Almer[1], Raymond[3]Osborn[5], and Michael Wilde[2,3]**

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The Swift parallel scripting language (http://swift-lang.org) allows APS users to perform large-scale concurrent execution of data analyses and simulation more efficiently and with less effort. At the APS, Swift is used to express and run analysis workflows for high energy diffraction microscopy (HEDM), diffuse scattering, and powder diffraction, as well as more general post-processing scripts of area-detector data. Recent use of Swift leveraged the ALCF Mira supercomputer for real-time analysis of a sample during an APS near-field HEDM experiment, allowing the detection and resolution of an apparatus error that would have otherwise gone undetected until much later. In such HEDM analyses, over 100,000 CPUs can be used concurrently. For *in situ* experiments where temperature and or strain is varied, fully automated Swift workflows can guide the experimental variables and eliminate manual errors, by analyzing in minutes what previously took months to perform without parallel processing. Furthermore, this allows users to leave the APS facility with most of their bulk data processing already completed.

The beamline user writes (or runs) what looks like ordinary serial scripts; Swift automatically spreads the work expressed in those scripts over parallel desktop, cluster, cloud or supercomputer resources. Swift efficiently automates functions which are hard, costly, and unproductive to do manually: 1) parallelization, using "data flow" techniques; 2) distribution of work and data across diverse systems; 3) failure/error handling; and 4) provenance recording for auditing, validation, and reproduction of results. As computing systems from laptops to supercomputers become increasingly parallel, Swift enables users to leverage this parallel power implicitly and largely automatically, with little or no experience in parallel programming.

# Polymers

A-55

## High Molecular Weight Insulating Polymers Can Improve the Performance of Molecular Solar Cells

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Solution-processed molecular semiconductors for the fabrication of solar cells have emerged as a competitive alternative to their conjugated polymer counterparts, primarily because such materials systems exhibit no batch-to-batch variability, can be purified to a greater extent and offer precisely defined chemical structures. Highest power conversion efficiencies (PCEs) have been achieved through a combination of molecular design and the application of processing methods that optimize the bulk heterojunction (BHJ) morphology. However, one finds that the methods used for controlling structural order, for example the use of high boiling point solvent additives, have been inspired by examination of the conjugated polymer literature. It stands to reason that a different class of morphology modifiers should be sought that address challenges unique to molecular films, including difficulties in obtaining thicker films and avoiding the dewetting of active photovoltaic layers. Here we show that the addition of small quantities of high molecular weight polystyrene (PS) is a very simple to use and economically viable additive that improves PCE. PS could give rise to film thickness and uniformity without sacrificing desirable phase separation and structural order. Remarkably, the PS spontaneously accumulates away from the electrodes as separate domains that do not interfere with charge extraction and collection or with the arrangement of the donor and acceptor domains in the BHJ blend.

A-56

## Dynamics of Nanoparticles within Gum Arabic Solutions

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Gum Arabic is a polysaccharide-protein complex widely used in food, pharmaceutical and cosmetic industries due to its good emulsifying, stabilizing and encapsulation properties. These technologically important features are related to the structural characteristics, conformation in solution and molecular composition of the biopolymer.

Herein we report x-ray photon correlation spectroscopy (XPCS) analysis using silica nanoparticles as a probe within dispersions of gum arabic from two different species. We found distinct intensity autocorrelation functions for each gum demonstrating that dynamics reflects proper characteristics of gum internal structure.

# Technique

A-57

## Tracking Molecular Dynamics from Picoseconds to Microseconds Using High-fidelity, Laser-pump, X-ray–probe Techniques

**Anne Marie March[1], Gilles Doumy[1], Elliot P. Kanter[1], Stefan Lehmann[1], Dooshaye Moonshiram[1], Stephen H. Southworth[1], Linda Young[1], Tadesse A. Assefa[2], Alexander Britz[2], Christian Bressler[2], Wojciech Gawelda[2], Zoltan Németh[3], and György Vankö[3]**

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Laser-pump, x-ray–probe techniques are powerful tools for exploring molecular structural changes that occur during a photo-initiated reaction. We are developing such methods at 7-ID-D of the Advanced Photon Source to study dynamics of molecules in solutions, combining x-ray emission spectroscopy and x-ray absorption spectroscopy as probes of electronic and geometric structure and using high-power, MHz lasers as pumps. The high-duty-cycle pump-probe measurements efficiently utilize the synchrotron x-ray flux and enable high-fidelity measurements of the structures of transient intermediates. Time scales from the ~80 picosecond x-ray pulse duration out to the microsecond regime can be explored. Recent measurements on solvated transition metal coordination complexes excited with 532 nm, 355 nm, and 266 nm laser light will be presented.

A-58

## Dioptas and T-Rax: New Python-based Programs for On-the-fly 2D X-ray Diffraction and Optical Spectroscopy Data Analysis

**Clemens Prescher and Vitali B. Prakapenka**

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Dioptas and T-Rax are open source Python programs with user-friendly graphical interface, designed for providing fast feedback during synchrotron and in-house experiments at ambient and extreme conditions.

Dioptas is intended to be a modern improvement of the widely used FIT2D software for analysis of x-ray powder diffraction data [1]. It uses the pyFAI library [2] as its basis for integration of 2D x-ray diffraction images and provides uniquely designed and easy to use interface for image auto-processing, browsing, detector geometry calibration and mask creation. It exhibits an optimized calibration algorithm with tunable parameters, which enables the calibration of even complex experimental geometries (high detector angle, off detector beam center position, etc.) [3]. The core of the application is a coordinated view where the image data and the integrated patterns can be explored simultaneously. Due to the fast integration time (~100 ms for a 2048 × 2048 pixels) collected data files can be investigated on-the-fly in almost real time. Integrated diffraction patterns can be compared by using scalable overlays and the positions of individual diffraction lines of selected (editable) phases can be displayed using the jcpds format. The line positions can be adjusted to pressure and temperature conditions if the equation of state parameters are provided. Dioptas includes a number of additional features for on-line or off-line data processing (e.g., algorithms for automatic background subtraction, and absorption correction for diamond and cBN seats typically used in diamond anvil cell experiments).

T-Rax is a graphical toolbox for the analysis of spectroscopic data frequently collected during high pressure diamond anvil cell experiments. It handles wide range of spectroscopic data measured with Princeton Instr. detectors (WinSpec or LighField file format) including thermal radiation, fluorescence and Raman spectroscopy. Temperature of laser or externally heated samples is calculated by fitting grey-body radiation curve to collected spectra with optional correction of the known system response. Unique features are automatic new file processing, fast switching between different system response calibrations and options, graphical selectable regions of interest, time-series analysis and communication with EPICS. Furthermore, T-Rax can calculate pressure at various temperatures using Ruby spectra, with optional peak fitting, and diamond anvil Raman spectra showing the derivative of the spectrum.

Dioptas has started being used for user operation at GSECARS/APS in June 2014 and is now frequently used/requested at other high-pressure beamlines, and T-Rax has been employed since September 2013.

[1] J.P. Hammersley, S.O. Svensson, M. Hanfland, A.N. Fitch, and D. Hausermann. *High Pressure Res.* **14:4-6**, 235–348 (1996).

[2] J. Kieffer and J. Wright. P*owder Diffr.* **28**, 339–350 (2013).

[3] C. Prescher and V.B. Prakapenka. *High Pressure Res.* Submitted.

## A-59

## Pink Beam Tomography at 13-BM-D

**Mark Rivers**

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Conventional monochromatic tomography at 13-BM-D covers the energy range from 6 to 60 keV with excellent energy resolution from a Si (111) monochromator. However, it is rather slow, requiring .25–5 seconds per projection, and hence 3 to 30 minutes per 3-D dataset. It allows performing above and below edge imaging, typically for elements like I, Cs, and Xe which are used as contrast agents for fluids in porous media.

We have now performed the first pink beam tomography measurements at 13-BM-D. Using pink beam reduces the exposure time to 1–5 ms, and the total time to collect a 3-D dataset to 7–12 seconds. This allows performing measurements on dynamic systems, albeit only rather slow ones.

The camera being used for both the monochromatic and pink beam measurements is a new CMOS camera from Point Grey that can collect up to 162 frames/s and costs less than $1,400.

## A-60

## Probing Optically Dense, Flowing Systems with X-ray Absorption and Fluorescence of Argon and Krypton at Beamline 7BM

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The non-invasive study of the internal structure of optically dense, flowing systems is challenging and requires techniques that can fully penetrate the system with high spatial resolution. Recently, two separate experiments were performed at Beamline 7BM using x-ray fluorescence to image and characterize flowing systems relevant to combustion research: a sooting flame and a resistively heated SiC micro-reactor. In both of these experiments, a 5 μm by 5 μm beam of x-rays was focused on the flowing system, and simultaneous x-ray fluorescence and absorption measurements were taken, with the fluorescence signal collected orthogonal to the incident x-rays. A 2D image was created by raster scanning across the flame or micro-reactor surface. The magnitude of the fluorescence signal is directly proportional to the density of fluorescing species and thus provides a way of measuring gas density in these systems. These measurements represent the first attempts to the authors' knowledge to probe gas density in high temperature, optically dense environments.

In the first experiment, an argon jet within a sooting atmospheric pressure flame on a modified McKenna burner was imaged with 8 keV x-rays. Flow rates of argon were varied to examine mixing of the argon jet with the flame gases and to estimate the sensitivity of the observation technique. The absorption and fluorescence measurements gave excellent agreement and the fluorescence technique gave better sensitivity. These experiments demonstrated that gaseous structures can be resolved with x-ray fluorescence and absorption with high spatial resolution within dynamic environments such as sooting flames. A second set of experiments were run after recent upgrades to Beamline 7BM which allowed absorption and fluorescence measurements with krypton. In these experiments, 15 keV x-rays were focused onto a resistively heated SiC micro-reactor, through which a few percent Kr in He was flowing. The x-rays penetrated the micro-reactor and were absorbed by Kr, and the resulting 12.6 keV fluorescence was detected. A 2D image of the micro-reactor was taken at different temperatures to map the density of Kr within the micro-reactor. Images of the free jet expansion of gases from the end of the micro-reactor into a region of relatively high vacuum were also taken. These data will be compared with CFD calculations of the flow within the micro-reactor in order to validate these calculations and guide further development and refinement of the CFD models. From there, realistic reaction conditions can be calculated thereby allowing accurate kinetic and mechanistic data to be obtained for pyrolysis and oxidation studies with SiC micro-reactors.

*This material is based upon work supported by the U.S. Department of Energy, Office of Science, Office Basic Energy Sciences, under Contract No. DE-AC02-06CH11357. This research used resources of the Advanced Photon Source, which is a DOE Office of Science User Facility.*

# 2015

# APS/CNM Users Meeting

# CNM POSTER ABSTRACTS

# Chemistry

## C-1

## X-ray Nanodiffraction Study of the Delithiation Mechanism of $LiFePO_4$ Single Particles

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Lithium iron phosphate ($LiFePO_4$) has received much interest as a cathode material on account of its good reversibility, low toxicity, thermal stability, and high material abundance [1]. However, there are some key fundamental drawbacks in that it is a poor ionic and electronic conductor, which are apparent in the rate performance. Understanding the intercalation mechanism will provide insight on some of the core limitations of this material. Two-phase and solid solution mechanisms have been proposed, but details on the transformation at the single particle level remain elusive [2]. X-ray nanodiffraction measurements on single particles were carried out to produce direct observations of the electrochemical reaction at a fundamental level.

Argonne National Laboratory's Advanced Photon Source boasts a hard x-ray nanoprobe beamline that has a very high spatial resolution of ~30 nm [3]. Traditionally, this beamline has been used to study thin films. Here, its power was harnessed to observe single microcrystals in a powder sample. Two-dimensional area maps were taken of one particle each of $LiFePO_4$, $FePO_4$, and an intermediate average powder composition $Li_{0.5}FePO_4$, which contained both fluorescence and diffraction data. Combining the fluorescence and diffraction data, the phases involved in the $LiFePO_4$-$FePO_4$ transformation were identified within the particle from the distance between lattice planes, calculated with Bragg's Law. Additionally, strain and inhomogeneities were also identified. Visualizing these phenomena on the single particle level and how they correlate to each other sheds light upon the electrode operation and how battery failure ultimately occurs. This information may lead to advancements in this material as well as those with similar structures.

[1] Gibot, P., Casas-Cabanas, M., Laffont, L., Levasseur, S., Carlach, P., Hamelet, S., Tarascon, J.-M., and Masquelier, C., *Nat. Mater.* **7** (9), 741–747, 2008.

[2] Meethong, N., Shadow Huang, H.-Y., Carter, W.C., and Chiang, Y.-M., *Electrochem. Solid-State Lett.* **10**, A134–A138, 2007.

[3] Winarski, R.P., Holt, M.V., Rose, V., Fuesz, P., Carbaugh, D., Benson, C., Shu, D., Kline, D., Stephenson, G.B., McNulty, I., and Maser, J., *J. Syn. Rad.* **19**, 1056–1060, 2012.

## C-2

## Conjugated Bridge Effects on Triplet Generation in Ladder-type Perylene Dimers

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Understanding the design of triplet generating chromophores is key to developing the next generation of optoelectronic organic materials. For example, long triplet exciton diffusion lengths can improve photocurrent in photovoltaic devices, and the generation of multiple triplet excited states from a single photon absorption event can potentially raise device efficiency above the Shockley-Queisser limit. We are investigating the photophysical properties of a series of ladder-type conjugated perylene diimide dimers using transient absorption spectroscopy on subpicosecond to microsecond timescales across the entire visible spectrum. These experiments show the generation of excited triplet states lasting tens of microseconds on the first of several molecules in the series. Here we investigate the effects of changes to the ladder-type bridge on the triplet generation properties of two such systems.

*We thank Dr. Dave Gosztola for help with transient absorption, fluorescence, and TCSPC experiments. Use of the Center for Nanoscale Materials was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.*

# Environmental Science & Geology

C-3

## Preliminary Assessment of Microstructures on Siliceous Microfossils in Subglacial Lake Whillans, West Antarctica

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The WISSARD Project recovered sediments from a subglacial lake beneath the Whillans Ice Stream which drains the West Antarctic Ice Sheet (WAIS) into the Ross Ice Shelf. Sediment samples from Subglacial Lake Whillans (SLW) provided the first look at a subglacial ecosystem that is isolated from the ocean and atmosphere. Analysis of the sediments revealed a thriving and diverse community of microbes such as extremophile bacteria, with a likely food source of fossil carbon and other nutrients derived from marine microfossils deposited during times when the ice sheet was in retreat and this was an open marine embayment. A variety of diatom ages are present, but the dominant age of the marine deposits is late Miocene (ca. 8 million years before present). The fossils include sponge spicules and diatoms.

We used the FEI Quanta 400F (E) SEM in association with the Center of Nanoscale Materials to analyze these fossils to document preservational effects of both subglacial mechanical shearing and microbial utilization of microfossil-based organic matter on the fossils. Mechanical degradation is evident by fresh fracture features, whereas microbial processes result in etching and pitting of the fossils. We find that diatoms are mechanically fragmented but lack dissolution features, whereas, sponge spicules have evidence of chemical etching features that we attribute to microbial attack, in addition to mechanical breakage. A microbial cell was observed on one of the spicules further supporting our hypothesis that the fossil sponge spicules provide a significant nutrient source for the microbial community beneath the WAIS.

# Materials Science

C-4

## Origins of Anisotropy in the Magnetic Structure of Artificial Spin Ice Lattices

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Artificial spin ice lattices are a two-dimensional analogue to spin ice crystals. The larger length scales of the artificial systems allow for their magnetic frustration behavior to be observed at room temperature. Traditionally, artificial spin ice lattices are patterned in thin films using electron-beam lithography because the elements that make up the lattice need to be small if they are to remain single domain. However, such fabrication is difficult to perform when the thin films have been deposited onto transparent membranes on TEM grids. Therefore, for those lattices whose elements remain single-domain at larger sizes, focused-ion beam (FIB) patterning represents a better alternative. In order to explore the origins of observed anisotropy in the lattices, Lorentz transmission electron microscopy has been used to obtain quantitative maps of the magnetization in square spin ice lattices FIB patterned in $Ni_{80}Fe_{20}$ (Permalloy) thin films. Our results have shown that although FIB patterning has an effect on the grain size of the Permalloy at the edges of the patterned regions, the anisotropy that is observed is the result of pre-existing anisotropy in the Permalloy film, rather being than an effect introduced by the FIB patterning. Our results thus suggest that FIB patterning is an effective method for fabricating larger-scale artificial spin ice lattices.

*This work was supported by the U.S. Department of Energy, Office of Basic Energy Sciences, Division of Material Sciences and Engineering. Use of the Center for Nanoscale Materials was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.*

## C-5

## Evolutionary Algorithm Search for Global Minimum Structures of Au Nano-clusters

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Gold nano-clusters are considered for applications such as optoelectronics, bio-recognition, and catalysis. In spite of their enticing potential, the atomic structures of very small (number of atoms <15) Au clusters are still elusive. For instance, there is ongoing debate on the critical cluster size, reportedly between 7 to 15 atoms, beyond which globular -3D- structures become more stable compared to planar -2D- ones. Furthermore, we found that some isomers at a given cluster size are energetically very close (<10 meV/atom), the reasons of which should be sought in finer details of electronic structures rather than mere differences in the number of dangling bonds at the edges or surfaces. Here, we present high throughput density functional theory calculations (DFT) coupled with a global structural optimization scheme using genetic algorithm (GA) to identify the ground state structures of Au nano-clusters near the critical cluster size. By investigating the electronic and vibrational properties of the low energy structures, we explore fundamental changes that drive the structural transformations.

## C-6

## Bond Order Potential to Capture Size-dependent Dimensionality Effects in Au Nanoclusters

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Gold nanoclusters, owing to their exceptional chemical, optical, and electronic properties, hold promise in a wide range of applications, such as optoelectronics, bio-recognition, and catalysis. Interestingly, Au nanoclusters are known to exhibit a diverse variety of structural configurations including planar, hollow cages, chiral tubes, and bulk-like pyramids or icosahedra. Despite these earlier observations, a fundamental understanding of the atomic structure of Au nanoclusters is still lacking — in particular, dependence of the structure of an $Au_n$ cluster on its size $n$ (i.e., number of atoms). For instance, there is an open debate on the critical cluster size $n_c$ beyond which globular isomers become energetically preferable over planar ones, with reported values of $n_c$ ranging from 7 to 15. Furthermore, the knowledge of structural transitions between energetically close isomers (~20 meV/atom) at a given size is still in its infancy. Global optimization and molecular dynamics (MD) simulations via empirical force fields (EFFs) provide an ideal route to address these issues. However, none of the available EFFs can adequately capture the dimensionality effects from bulk to sub-nanometer clusters. Popular pairwise Au EFFs, such as Morse, or those that account for multi-body effects via an embedding function (e.g., EAM, MEAM) tend to artificially over-stabilize bulk-like isomers. Here, we employed genetic algorithms to parameterize a bond-order potential based on Tersoff functional form by training against cohesive energies of representative clusters and condensed bulk phases of Au computed by density functional theory (DFT) calculations. This new EFF was found to be capable of describing the diverse range of structures of Au nanoclusters consistent with previous reports (e.g., planar structures circa $Au_{13}$, hollow cage at $Au_{14}$, and compact pyramid at $Au_{20}$). Furthermore, we employed long-time MD simulations with this EFF to identify the mechanisms governing agglomeration of clusters Finally, our new hybrid EFF, which can describe Au clusters well in the nanometer length scale; this will open doors to structure prediction of technologically relevant mid-size Au clusters (n=20—80) that are not tractable by DFT alone.

C-7

## $IrO_2$ Surface and Nanostructure Stability from First Principles and Variable Charge Force Field Calculations

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$IrO_2$ is one of the most efficient water-oxidation electrocatalysts and can also act as a photocatalyst for the water splitting reaction when used in the form of nano-sized clusters. The structure and surface properties of nanoclusters greatly influence the photocatalytic properties of $IrO_2$, and atomistic scale understanding of these properties is essential to elucidate the photocatalytic mechanisms of $IrO_2$. Here, we carried out first principles calculations based on spin polarized density functional theory (DFT) including spin-orbit coupling and the Hubbard $U$ correction on the bulk and surface structures of $IrO_2$. The stability and electronic structure of low index rutile (100), (001), (110) and (101) surfaces of $IrO_2$, were studied. The relative surface energies were obtained as (110) < (101) < (100) < (001). The equilibrium shape of $IrO_2$ nanoparticles was deduced using a Wulff construction. In order to study the structural stability of $IrO_2$ nanoclusters and the long time-scale dynamics of $IrO_2$ polymorphs larger than about 2 nm in diameter, we developed the first empirical interatomic potential (force field) for $IrO_2$ based on Morse functional form coupled with a variable charge method (QEq). The Morse+QEq parameters were optimized using an evolutionary algorithm with respect to a DFT training set, and was shown to be successful in predicting bulk and surface properties of rutile $IrO_2$ and various polymorphs including anatase, brookite, columbite and pyrite derived from DFT calculations. Pressure induced phase transformations of bulk $IrO_2$ polymorphs were reported and thermodynamically stabile phases of $IrO_2$ at nano-scale were obtained using the surface energies of stable polymorphs. Our results will shed light on the development of stable nanoscale $IrO_2$ electrocatalysts and photocatalysts that can efficiently utilize solar energy for water splitting reaction.

# Nanoscience and Nanotechnology

C-8

## New Fabrication Techniques for Cryogenic Micro-calorimeters

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Cryogenic micro-calorimeters provide the highest energy resolution of any solid state x-ray detectors. At APS we have been focused on developing a superconducting technology called thermal kinetic inductance detectors (TKIDs), which is a resonator-based technology. We present the results of several new fabrication efforts at CNM to increase the performance of TKIDs: new alloys of WSix with tunable Tc from 500mK to 5K, slopped SiN etching via resist reflow, and deep silicon etching of x-ray apertures. These techniques allow us to reduce thermal fluctuation noise due to a lower Tc resonator material, reduce TLS noise by removing the SiN underneath the resonator capacitor, and limiting spurious x-ray pulses by blocking x-ray absorption in the device wiring with the use of the aperture.

C-9

## Lithium Sulfur Batteries as Energy Storage Devices

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The lithium sulfur battery as an excellent alternative for energy storage devices has attracted extensive interests. Herein, we adopted a facile approach to synthesize the $Li_2S$ @ nitrogen-doped carbon, which contains the active

materials as high as 72%. With SEM and EFTEM characterizations, the size of this composite is about 400 nm, and has a very great core-shell structure for protecting lithium polysulfides from dissolutions to electrolytes. The doped nitrogen revealed by XPS facilities electrons diffusion in the battery system and thus enables a superior electrochemical reaction condition. The $Li_2S$ encapsulated by nitrogen-doped carbon can deliver extremely high specific capacity of 1029 mA h/g at 0.2 C and retain 652 mA h/g even after prolonged 100 cycles.

C-10

## ZnO Functionalization of Surface Pre-treated Multi-walled Carbon Nanotubes for Methane Sensing

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Bare carbon nanotubes (CNTs) are insensitive towards most gases due to poor bonding between the chemically inert graphitic surface and different compounds they are exposed to. Consequently, for gas sensing applications, functionalization of CNTs with reactive compounds is required. By introducing surface pre-treatments prior to functionalization, the affinity of the functionalizing species is enhanced, enabling the fabrication of highly sensitive CNT chemiresistor-based sensors.

Atomic layer deposition (ALD) allows precise, uniform and conformal deposition of oxide coatings on geometrically complex substrates such as MWCNTs [1]; thus offering a suitable route for the functionalization of MWCNTs for gas sensing applications. Motivated by the energetically favorable electron transport in ZnO-MWCNT junctions [2], we have performed ALD of ZnO for functionalizing O2 plasma and UV-O3 treated MWCNTs. Diethylzinc ((C2H5)2Zn) was used as an ALD precursor. Deposition was performed at three different temperatures, 175,200 and 225°C, with an Arradiance Gemstar ALD tool. Transmission electron microscopy (TEM) images show uniform deposition of ZnO nanoparticle (NP) layers on the MWCNTs. At ALD temperature of 175°C and 200°C the average NP size was found to be 7.2 nm (standard deviation, SD, 1.18 nm) and 10.8 nm (SD 1.81 nm) respectively. The higher resolution TEM image illustrates the wurtzite structure of the ZnO-NP and its good crystalline quality. The interplanar spacing of 2.8Å, 2.68Å and 2.48Å correspond to <100>, <002> and <101> planes of ZnO [3]. Raman spectroscopy also suggests good crystal quality of ZnO-NP. The chemiresistor sensors based on ZnO functionalized MWCNTs were used to detect ppm level concentrations of methane in zero air at room temperature.

[1] S. Boukhalfa, K. Evanoff, and G. Yushin, *Energy & Environment Science*, 2012, **5**, 6872.

[2] H. Zhang, N. Du, B. Chen, D. Li, and D. Yang, *Science of Advanced Materials,* 2009, **1**(1), 13–17.

[3] P. Bindu and Sabu Thomas, *Journal of Theoretical & Applied Physics*, 2014, **8**:123–134.

*Use of the Center for Nanoscale Materials, Argonne National Laboratory was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357. The project is in part funded by the College of Engineering, University of Illinois, Chicago, IL.*

## C-11

# Nanostructure Electrodes for Perovskite Solar Cells and Electrochromic Devices

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Charge transport at the interfaces and bulk phases governs the efficiency of various heterojunction optoelectronic devices such as electrochromic cells and solar cells. Here, I will present my research works on charge transport processes in two different optoelectronic devices: nanostructured heterojunction photovoltaic cells and electrochromic devices.

Current lead halide perovskite solar cells have photoactive layer less than 600 nm due to the increment of dark current and electron transport resistance in thicker layer. $TiO_2$ nanowires have been used in perovskite solar cells and photoactive layer is as thick as 900 nm. We apply these rutile NW arrays with different length as photoanodes in perovskite solar cells and we achieved 11.7% efficiency, which is 2% (absolution value) higher than the best perovskite solar cells using nanowire as photoanode reported in the literatures [1].

Lead halide perovskite solar cells use low chemical potential, that is, high work-function (ϕ) precious metals, such as gold (ϕ=5.1 eV), as the back cathode to maximize the attainable photovoltage. We report herein a set of perovskite type solar cells that use nickel (ϕ=5.04 eV), an earth abundant element and non-precious metal, as back cathode, and achieve nearly the same open-circuit voltage as gold and an efficiency of 10.4%. This work opens a “nickel” -and-dimed (low-cost) way towards high-efficient perovskite solar cells [2].

A solid-state electrochromic (EC) device based on 3-D conductive fluorinated tin oxides (FTO) nanobeads electrodes with over 500 roughness factor was obtained. The 3-D conductive FTO nanobeads electrode reduced the driving voltage to less than 1.2 V and response time to 272 ms, in comparison 2-D flat FTO film as the electrode [3].

[1] Jiang, Q.; Sheng, X.; Li, Y.; Feng, X.; and Xu, T., Rutile $TiO_2$ nanowire-based perovskite solar cells. *Chem Commun (Camb)* 2014, **50** (94), 14720–14723.

[2] Jiang, Q.; Sheng, X.; Shi, B.; Feng, X.; and Xu, T., Nickel-Cathoded Perovskite Solar Cells. *The Journal of Physical Chemistry C* 2014, **118** (45), 25878–25883.

[3] Jiang, Q.; Liu, F.; Li, T.; and Xu, T., Fast and low voltage-driven solid-state electrochromics using 3-D conductive FTO nanobead electrodes. J*ournal of Materials Chemistry C* 2014, **2** (4), 618.

## C-12

# Sequential Infiltration Synthesized ZnO Nanostructures

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We present a method of creating zinc oxide (ZnO) nanostructures that may be used for photonic applications by combining lithography and an ALD process named Sequential Infiltration Synthesis (SiS) [1]. The SiS method utilizes similar concepts of ALD with the significant difference in process exposure times, pressure, and purpose. The purpose is to allow the precursor gases infiltrate a polymer matrix (e.g., polymethyl methacrylate or PMMA) and allow the reaction to occur inside the polymer matrix. To achieve this it is necessary to allow time for the gases to diffuse (longer exposure times and higher pressures). Although SiS has been used mainly for applications with block copolymers [1], it can be also used in conjunction with lithography.

We demonstrate that SiS process of ZnO allows the formation of ZnO throughout 270 nm of PMMA and shows we can create unusual ZnO nanostructures by combining ZnO SiS process with lithographically patterned PMMA. X-ray and photoluminescence data of SiS treated PMMA will be presented.

*Use of the Center for Nanoscale Materials was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.*

[1] Q. Peng, Y.-C. Tseng, S.B. Darling, and J.W. Elam, *ACS Nano*, 2011, **5** 4600.

## C-13
## Development Characteristics of Polymethyl Methacrylate in Alcohol/Water Mixtures

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Poly methyl methacrylate (PMMA) is the most widely used resist in electron beam lithography. This paper reports on the study of development characteristics of PMMA in methanol, ethanol and isopropanol (IPA) mixtures with water as developers. We have found that ethanol/water mixtures at a 4:1 volume ratio are an excellent, high resolution, non-toxic, developer for exposed PMMA and GL-2000 resist. Ethanol is much less hazardous than both methyl isobutyl ketone (MIBK) and IPA (traditional PMMA developers). As a developer, ethanol had been used in combination with other solvents [1] or as "95%" ethanol [2,3]. No detailed study of ethanol-water mixtures as a developer for PMMA has been previously reported as far as we have investigated.

Our results show how powerful simple lithographic techniques can be used to study ternary polymer solvent solutions when compared to other techniques found in the literature. The impact of the understanding these interactions may open doors to a new family of developers for other electron beam resists that can reduce the toxicity of the waste stream. We have already tested Ethanol:Water in 4:1 volume ratio on GL-2000-12 resists (similar to ZEP 520A) with good resolution. Contrast, resolution and Raman spectral data will be presented.

*Use of the Center for Nanoscale Materials was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.*

[1] W.J. Cooper, P.D. Krasicky, and F. Rodriguez, *J. of Applied Polymer Science* 1986, **31**, 65.

[2] R. Shimizu, T. Ikuta, T.E. Everhart, and W.J. DeVore, *J. of Applied Physics* 1975, **46**, 1581.

[3] D.F. Kyser and N.S. Viswanathan, *J. Vac. Sci. Technol.* 1975, **12**, 1305.

## C-14
## Study of Polarization Anisotropy of Grown Cadmium Sulphide Nanowires

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Cadmium sulphide (CdS) nanowire arrays were grown using a nanoporous template via electrodeposition. Raman and PL intensity were recorded for parallel and perpendicular polarization with two orientations of the sample having light propagating parallel to the nanowire axis in one orientation and light propagating perpendicular to the nanowire axis in other orientation. Furthermore, to study orientation dependent electron phonon (EP) coupling polarized Raman spectroscopy was also performed on wurtzite CdS nanowires for different angles of the axis of nanowire (z axis) and the laser polarization. Strong peaks of different phonon modes belonging to wurtzite structured CdS nanowires were observed with different polarizations of Raman spectral studies.

The EP coupling strength is estimated by the ratio of Raman intensity of the 2 longitudinal optical (LO) phonon peak with respect to that of the 1 LO phonon peak, according to the Franck Condon approximation. Therefore, the strongest EP coupling was observed when the polarization of the laser is parallel to the axis of the nanowire, and decreases until the polarization of the laser is perpendicular to the axis of the nanowire. The Hamiltonian function of EP coupling strength for wurtzite CdS crystals was computed for the different angles between the long axis of nanowire (z axis) and the laser polarization for comparison with experimental observations. The computed EP coupling constant decreases with the increasing angle between the axis of nanowire (z axis) and the laser polarization as observed experimentally. The Raman and photoluminescence (PL) properties were characterized for the grown CdS nanowires which showed a remarkable polarization anisotropy of 0.80 in the PL intensity, portending the possible use of these nanowire structures in polarization-based sensitive nanoscale devices for optoelectronic applications.

C-15

## Fabrication of Large Transition Edge Sensor Bolometer Arrays for CMB Measurements in the Upcoming SPT-3G Experiment

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Since its discovery, the cosmic microwave background (CMB) has played a pivotal role in cosmology and particle physics. The ability to make precision measurements of the CMB is led by detector innovation. The frontier of CMB detector development is focused on improving bolometric detectors based on superconducting Transition Edge Sensors (TES). In this sense, this work presents the fabrication of large arrays of multi-chroic TES bolometers for CMB measurements using the South Pole Telescope. The resulting array of detectors, known as SPT-3G, will be the third generation CMB camera to be installed in the South Pole Telescope. It will deliver a factor of ~20 improvement in mapping speed over the current receiver, SPT-POL.

Each pixel is composed of a broad-band sinuous antenna coupled to a niobium microstrip line. In-line filters are used to define the different band-passes before the millimeter-wavelegth signal is brought to the respective Ti/Au TES bolometers. There are six TES detectors per pixel, which allow for measurements of three band-passes (90 GHz, 150 GHz and 220 GHz) and two polarizations. When finished, the SPT-3G camera will be composed ~2700 pixels, featuring a total of ~16000 TES bolometric detectors. The steps involved in the monolithic fabrication of these detectors arrays are presented in detail in this work. Patterns are defined using a combination of stepper and optical lithography. The misalignment between layers is kept below 200 nm. The overall fabrication process involves a total 16 steps, which include PECVD deposition, reactive and magnetron sputtering, RIE, RIE-ICP and chemical etching. To assure a good performance of the detectors, the quality of the metallic and dielectric films involved in the fabrication is controlled using both cryogenic and room temperature testing.

*Work at Argonne National Laboratory, including the work at the Center for Nanoscale Materials, a U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences User Facility was supported by the Office of Science and Office of Basic Energy Sciences of the U.S. Department of Energy, under Contract No. DE-AC02- 06CH11357. The South Pole Telescope is supported by the National Science Foundation through grant PLR-1248097. Partial support is also provided by the NSF Physics Frontier Center grant PHY-1125897 to the Kavli Institute of Cosmological Physics at the University of Chicago, the Kavli Foundation and the Gordon and Betty Moore Foundation grant GBMF 947.*

## C-16

## Spectroscopic Imaging of NIR to Visible Upconversion from $NaYF_4$: $Yb^{3+}$, $Er^3$ Nanoparticles on Au Nano-cavity Arrays

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We use spectroscopic imaging to assess the spatial variations in upconversion luminescence from $NaYF_4$:$Er^{3+}$,$Yb^{3+}$ nanoparticles embedded in PMMA on Au nano-cavity arrays over a wide range of excitation intensities. The nano-cavity arrays support a surface plasmon (SP) resonance at 980 nm, coincident with the peak absorption of the $Yb^{3+}$ sensitizer. Spatially-resolved upconversion spectra show a 30X to 3X luminescence intensity enhancement on the nano-cavity array compared to the nearby smooth Au surface, corresponding to varying excitation intensities from 1 W/cm$^2$ to 300 kW/cm$^2$, spanning the non-linear and saturation power dependence regimes. Our analysis shows the power dependent enhancement in upconversion luminescence can be almost entirely accounted for by a constant shift in the effective excitation intensity, which is maintained over five orders of magnitude variation in excitation intensity. The variations in upconversion luminescence enhancement with power are modeled by a 3-level-system near the saturation limit, and by simultaneous solution of a system of coupled nonlinear differential equations, both analyses agree well with the experimental observations. The amplification of the excitation field is independent of the emission wavelength, suggesting the enhancement in upconversion emission is due to entirely increased absorption by the $Yb^{3+}$ sensitizer. Analysis of the statistical distribution of emission intensities in the spectroscopic images on and off the nano-cavity arrays provides an estimate of the average enhancement factor independent of fluctuations in nano-particle density.

## C-17

## Frictional Properties of Graphene on Silica Surfaces with Nanoscale Roughness

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Microelectromechanical systems (MEMS) devices suffer from severe wear and short lifetimes from high pressure asperity-asperity contacts due to the nature of their rough surfaces. Recently nanoscale carbon lubricants, such as graphene, have become of interest as protective surface coatings for MEMS. Here, AFM has been used to investigate the frictional properties of graphene on hydrophilic and hydrophobic nanoparticle films, which model the nanoscaled asperities found on realistic surfaces. Combined AFM and Raman microspectroscopy studies revealed that graphene is strained and partially conforms to the rough surfaces. As the number of layers increase, conformity decreases due to the bending stiffness, but increases under mechanical loading. Friction nominally decreases as a function of layer thickness, but was also found to depend on contact area of the tip and interfacial shear strain of the graphene associated with its adhesion to the substrate. Adhesion of graphene to the tip was found to depend on the degree of surface roughness and humidity.

## C-18

## Novel Approaches towards Efficient Mg Rechargeable Batteries

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To meet surging demands for sustainable energy and clean environment, one critical requirement is to develop high-energy, safe and low-cost rechargeable batteries for electric transportation and grid energy storage. Mg rechargeable batteries (MgRBs) stand out as a promising candidate beyond lithium ion battery technologies due to high volumetric energy density, resource abundance, and the dendrite-free deposition behavior of Mg, which ensures

safe operation [1]. Many of the advantages of MgRBs originate from the divalent nature and small ionic size of Mg ions; however, these properties also render the cation too polarizing to diffuse easily in most ion-intercalation materials. We recently investigated interlayer expansion as a general and effective atomic-level lattice engineering approach to transform inactive layered intercalation hosts into efficient Mg storage materials without adverse side effects [2]. We have combined theory, synthesis, electrochemical measurements, and kinetic analysis to improve Mg diffusion behavior in MoS2, which is a poor Mg transporting material in its pristine form. The expansion boosts Mg conductivity by two orders of magnitude, effectively enabling the otherwise barely active $MoS_2$ to approach its theoretical storage capacity as well as to achieve one of the highest rate capabilities among Mg-intercalation materials. The interlayer expansion approach can be leveraged to a wide range of host materials for the storage of various ions, leading to novel intercalation chemistry and opening up new opportunities for the development of advanced materials for next-generation energy storage.

In addition, we also demonstrated the excellent stability of a high areal capacity hybrid magnesium-lithium-ion batteries (MLIBs) cell and dendrite-free deposition behavior of Mg under high current density [3]. The hybrid cell showed no capacity loss for 100 cycles with Coulombic efficiency as high as 99.9%, whereas the control cell with a Li-metal anode only retained 30% of its original capacity with Coulombic efficiency well below 90%. The use of $TiS_2$ as a cathode enabled the highest specific capacity and one of the best rate performances among reported MLIBs. Postmortem analysis of the cycled cells revealed dendrite-free Mg deposition on an Mg anode surface, while mossy Li dendrites were observed covering the Li surface and penetrated into separators in the Li cell. The energy density of a MLIB could be further improved by developing electrolytes with higher salt concentration and wider electrochemical window, leading to new opportunities for its application in large-scale energy storage.

*Acknowledgment: We thank the funding support from the U.S. Office of Naval Research (No. N00014-13-1-0543).*

[1] Aurbach, D., Lu, Z., and Schechter, A., et al. Prototype systems for rechargeable magnesium batteries. *Nature*, 2000, **407**, 724–727.

[2] Liang, Y., Yoo, H.D., and Li, Y., et al. Interlayer-Expanded Molybdenum Disulfide Nanocomposites for Electrochemical Magnesium Storage. *Nano Letters*, 2015, **15**, 2194–2202.

[3] Yoo, H.D., Liang, Y., and Li, Y., et al. High Areal Capacity Hybrid Magnesium-Lithium-Ion Battery with 99.9% Coulombic Efficiency for Large-Scale Energy Storage, *ACS Applied Materials & Interfaces* DOI: 10.1021/acsami.5b01206.

## C-19

## Protection of Exfoliated Black Phosphorus Transistors from Ambient Degradation

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Black phosphorus (BP), a two-dimensional phosphorus allotrope, is unique among layered nanomaterials due to its anisotropy, high carrier transport (~1000 $cm^2V^{-1}s^{-1}$), tunable band gap (~0.3 to 1.5 eV), and chemical reactivity. However, the reactivity of BP leads to its chemical degradation in ambient conditions, affecting high performance applications utilizing the nanomaterial. When unencapsulated, exfoliated BP flakes degrade into oxidized phosphorus compounds, as determined by atomic force microscopy, x-ray photoelectron spectroscopy, Fourier transform infrared spectroscopy, transmission electron microscopy, and electrostatic force microscopy measurements. Oxygen-saturated $H_2O$ is a major source of this BP oxidation, since BP flakes placed on hydrophobic substrates are observed to degrade two times faster than on hydrophilic substrates. After 48 hours in ambient, unencapsulated BP field-effect transistors (FETs) decrease in mobility and current on/off ratio by factors of 1000. Conversely, $AlO_x$ encapsulated BP FETs do not oxidize, maintaining mobilities of ~100 $cm^2V^{-1}s^{-1}$ and on/off ratios of 1000 for over four months in ambient. This strategy to protect BP against ambient oxidation will facilitate the realization of BP electronic and optoelectronic applications and augment ongoing fundamental BP research [1].

[1] J.D. Wood et al., *Nano Lett.* **14**, 6964 (2014).

C-21

## X-ray Absorption Spectroscopy (XAS) Study of Sorption Mechanisms of Cd(II) to Hematite ($\alpha$-$Fe_2O_3$) Nano Particles with Varying Size and pH Environment

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Natural mineral nanoparticles have gained attention due to their wide occurrence in soil and high sorption capacities for cationic and anionic contaminants influencing the mobility and transport of contaminants in the environment. This is a consequence of their small size and non-optimal surface coordination environment compared to the bulk. Hematite (alpha-$Fe_2O_3$) is a mineral whose particles are found in the nano-sized region. This study involves investigating the sorption mechanisms of the highly toxic heavy metal cadmium to hematite. Experiments were normalized to total hematite nanoparticle surface area within reaction vessels. The sorption mechanisms were investigated under different hematite particle sizes (8nm and 40nm surface area/mass normalized (SAN/MN)) and different pH environments (pH 7.5 and pH 9).

The adsorption edge experimental results suggested particles sorbed more Cd(II) as the pH environment was increased and the particle size was decreased. For the larger particles, the sorption edge was shifted to the right approximately by 1 pH unit. X-ray absorption near edge structure (XANES) results on 8 nm nanoparticles and the 40 nm SAN at pH 7.5 indicated the presence of similar coordination environment around the absorbing Cd atom. Cd was adsorbed to 8 nm particles at pH 7.5 but did not form a precipitate whereas at pH 9, minor amounts of $CdCO_3$ and CdO were present. When experiments were normalized to hematite surface area, particle size did not substantially affect the sorption mechanism at pH 7.5. However, at pH 9 a combination of $CdCO_3$ precipitate and adsorption complex(es) were formed of which contribution from the precipitate was larger. When experiments were normalized to hematite mass, more Cd precipitation was observed in larger nano particles at both pH values. This is most likely due to the presence of fewer surface adsorption sites on the particles. Extended x-ray absorption fine structure (EXAFS) results revealed binding site details as a function of pH and particle size and will be presented.

Application of XAS technique for the purpose of gaining insights into the mechanisms of interaction between hematite nano particles and heavy metal cadmium suggests a first step towards alleviating nano particle related environmental and health hazards.

C-22

## Integrated Photonics with Single-layer $MoS_2$

**Guohua Wei[1], Teodor Stanev[2], David Czaplewski[3], Il Woong Jung[3], and Nathaniel P.Stern[1, 2]**

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Two-dimensional transition metal dichalcogenides (TMDCs) such as $MoS_2$ are direct band gap semiconductors at the limit of atomic-scale thickness. The strong optical transitions across the direct band gap of monolayer TMDCs suggest that atomically thin 2D semiconductors can be useful materials for interfacing with photonics to achieve new device functionality. We report on the integration of $MoS_2$ monolayers with silicon nitride microresonators assembled by visco-elastic layer transfer techniques. Coupling between the photonic mode and the monolayer semiconductor flakes is confirmed by reduction of the quality factor. This achievement paves the way for functional integrated optoelectronic devices such as modulators that harness the optical properties of 2D semiconductors.

# Technique

C-20

## Microfabrication of All-copper Waveguides by UV-lithography

**Olga V. Makarova[1], Ralu Divan[2], and Cha-Mei Tang[3]**

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In order for radar systems to see through clouds at video frame rates, the frequency of transmission must be much higher than radio frequency, and the transmission power must be high to enable video rate. As frequency increases, waveguide feature structure dimensions and tolerances decrease. Submicron precision and surface finish are needed in order to achieve the required power performance of the circuits. Thermal management of high radiation power is needed for stabile continuous operation, which can only be achieved with an all-copper structure for the waveguide.

We report the fabrication of two-level all-copper folded waveguide circuits. The fabrication method is based on UV-lithography using SU-8 negative photoresist, followed by copper electroforming. The method achieved the necessary dimensional accuracy, smooth vertical side walls, low fabrication cost, and all-copper structure suitable for high power applications.

*Use of the Center for Nanoscale Materials, Argonne National Laboratory was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.*

# 2015

## APS/CNM Users Meeting

# ESRP POSTER ABSTRACTS

# Exemplary Student Research Program

## ESRP-1

## Nanoscale Elemental Fingerprinting of Historic Ink

**Riley Brooksher[1], Kyle E. Jensen[1], Andrew T. Li[1], Andrew Z. Shao[1], Volker Rose[2], and Katherine Seguino[1]**

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Historic inks from a wide range of dates, from as far back as the 5th century, have shown signs of corrosion and degradation. Research into these inks can lead to progress in preserving important handwritten historical documents. We have utilized high-resolution x-ray fluorescence to analyze the elemental composition of a sample of mid-19th century ink. Additionally, the physical characteristics of the paper were analyzed by an optical light microscopy. Optically, torn fibers and vertical ink displacement were observed. Through nanoscale x-ray fluorescence concentrations of each element were identified and compared. The x-ray fluorescence maps indicated the presence of certain key elements in the ink but not paper: namely iron, calcium, nickel. Metallic atoms organized themselves into nanospheres and other organized distributions while metalloids such as Al, Si, and Mn are uniformly distributed. Notable variations include the decrease in chlorine in the sample with ink.

*This research was supported through the Exemplary Student Research Program, supported by Argonne National Laboratory Educational Programs (CEPA), Naperville Central teacher, Katherine Seguino, and supported by the Advanced Photon Source and the Center for Nanoscale Materials APS is supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract DE-AC02-06CH11357. The authors thank the Naperville Heritage Society for providing historic samples.*

## ESRP-2

## Sequestered Elements in Tissues of Water Willow and Freshwater Mussels as a Function of Sustainable Urban Stream Ecology

**Karen Beardsley[1], Samuel Carani[1], Megan Gerbuyos[1], Hunter Easterday[1], Tyler Huber[1], Aime Luna[1], Judy Nguyen[1], Nicholas Scipione[1], and David Vine[2]**

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The ecological health of urban streams has recently gained interest alongside the increased spread of suburban and urban communities. Urban streams commonly exhibit eutrophication as a result of increased nutrient load from point and nonpoint sources in the local communities. Point sources, such as wastewater treatment plants, contribute to high nutrient load and eventual cultural eutrophication of these ecosystems by releasing increased amounts of nitrates and phosphates. The Illinois EPA has determined the Spring Brook creek of Wheaton, IL to be impaired. This rating has resulted in a stream mitigation plan prepared by the DuPage Forest Preserve District's Urban Streams department. Spring Brook creek restoration plans by the DuPage Forest Preserve District include building a more natural streambed and the introduction of organisms that together will result in more sustainable stream ecology. The stability of the streambeds and development of sustainable microhabitats will be improved by the introduction of the plant American Water Willow (*Justicia americana (L.) Vahl.)*. It is not known if Water Willow is a bio-acculmulator. If Water Willow sequesters phosphorus and metals, this process would contribute to improvement of the stream ecology. The introduction of freshwater mussels, Giant Floater, *Pyganodon* (=*Anodonta*) *grandis* and White Heelsplitter, *Lasmigona complanata,* will occur in the second phase of the stream restoration. Freshwater mussels are part of and serve as an indicator of healthy streams. They provide microhabitats for various species, filter water, and sequester metals. Due to certain urban environmental stressors, it has been estimated that urban streams have lost a majority of our indigenous mussel species.

This research hopes to determine the possible contribution of Water Willow toward reducing stream water nutrient load by detection of plant tissue sequestration of phosphorus and metals. The measurement of phosphorus and metal composition in Giant Floater and White Heelsplitter tissue will serve as baseline data regarding the possible mussel contribution toward maintenance of stream ecosystem stability.

ESRP-3

## X-ray Crystallography of IF7 and Concanavalin-A

**Kathleen Dwyer, Hooi Nee Kee, Paul Meuser, Natalie Mitten, Zaphron Richardson, Mia Rintoul, and Anna Wermuth**

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The development of vascular supply is an essential source of the growth and metastasization of malignant tumors. Annexin 1, a potential anti-cancer drug involved in human anti-inflammatory processes, is capable of highly specific tumor vasculature recognition. Research has identified the carbohydrate ligand-mimicking 7-mer peptide, (IFLLWQR or IF7) as capable of targeting Annexin A1 in mouse tumors. IF7 has exhibited unprecedented tumor-targeting activity and has been detected in mouse tumors within a few minutes of intravenous injection of the peptide [PNAS, 108(49),19587-19592 (2011)]. IF7 has the potential to act as a vehicle for anticancer drugs delivering them to the location of the tumor. Concanavalin-A is a carbohydrate-binding lectin, originally extracted from Jack Bean *Canavalia ensiformis*.

It binds to various sugars, glycoproteins and glycolipids, by recognition of a Î±-D-mannosyl or a Î±-D-glucosyl group. In this experiment, crystals of Concanavalin-A were grown, cross-linked with glutaraldehyde, then soaked in solutions of IF7 and IF7CRR, to identify their biologically active conformation.

Co-crystals of the protein/peptide complexes were also grown, independently of the glutaraldehyde cross-linking process and crystals of the IF7 and IF7CRR peptides. Diffraction images were collected using Beamline 19 at Argonne National Laboratory and the results were analyzed to determine the macromolecular structure of the crystalline contents.

ESRP-4

## The Effect of Increased Phosphorous on Metal Absorption in Lettuce

**Austin Balinski[1], Brett Cohen[1], Lydia Finney[2], Thomas Matysik[1], Theresa Quain[1], Anne Rock[1], Yvonne Szustakiewic[1], Rachele Wen[1], April Wendling[1], and Eric Wynne[1]**

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Varying levels of phosphorus will be added to separate samples of lettuce plants. In addition to the phosphorus, a controlled amount of nickel, zinc and iron in solution will be added to all the samples. The metal concentrations and distribution in the leaves will be analyzed using the APS. Since phosphorus is linked to root growth, we expect that the phosphorus should increase the amount of metal found in the lettuce leaves. Some researchers have found that high levels of phosphorous prevent metal uptake. We expect that at a certain point, the effects of adding phosphorus in relation to metal absorption would reach a peak. After this point, increasing the levels of phosphorus would no longer have a positive correlation with the metal absorption. By finding the range of peak metal absorption, we may be able to determine the optimal amount of phosphorus. The results will give us more insight on the effects of adding certain aqueous solutions during the plant's growth on leaf growth and metal absorption. To find these results, we will need to read and accurately interpret the levels of metal absorption found within the varying phosphorus sampled plants.

ESRP-5

## Understanding the Reduction Mechanisms and Structural Changes of a Lithium-rich Oxide Material Using *Operando* X-ray Absorption Spectroscopy

**Jason Croy[2], Deepankar Gupta[1], Dominica Lange[1], Logan Mazurek[1], Daria Prawlocki[1], Jeremy Shin[1], Casey Stowers[1], Jack Thomas[1], and Linda Wilkins[1]**

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Taking advantage of the element specificity and operando capability of x-ray absorption spectroscopy, we have studied the evolution of the local electronic and atomic structure of a lithium-rich oxide material, $0.1Li_2MnO_3$ $0.9LiCoO_2$, during electrochemical discharge in a half-cell. The study elucidates the mechanism of the reduction reaction of this complex material and sheds light on the nature of the reduced species generated during the lithiation reaction. The findings of this study help enhance our understanding of conversion and alloying reactions. Additionally, these studies are of relevance to the topic of performance degradation — initiated by a crosstalk between the positive and negative electrodes — in lithium-ion full cells.

ESRP-6

## Determining the Suitability of Fly Ash Produced in Illinois for Use in Geopolymer Concrete

**James Birrell[1], Kevin Conway[2], David Kavalauskas[1], Brenden Kelly[1], Elizabeth Klimek[1], Joe Muller[2], and Abigail Zatkalik[1]**

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This proposal hopes to determine the suitability of locally produced fly ash for use in construction-grade materials. Fly ash is a by-product of coal burning power plants, and 4.4 million tons of fly ash are produced annually in Illinois alone. Fly ash is known to contain many harmful elements, and disposal of fly ash has historically been problematic. One possible route to beneficiation of fly ash is to use it as a geopolymer concrete. However, the question remains as to whether or not this geopolymer concrete is environmentally safe. We hope to determine the answer to this question by using fly ash generated or used in Illinois and analyzing the sample for the presence of Cr(VI) and other harmful elements. By determining both the concentration of potentially hazardous elements and their spatial distribution in the sample, we hope to be able to reach some conclusion regarding the potential environmental impacts of the use of locally produced fly ash as a geopolymer concrete.

ESRP-7

## Comparing Extant and Fossilized Spiders to Determine Evolutionary and Preservational Changes Using Synchrotron X-ray Tomography

**Ahmad Abdulla[1], Christopher Figus[2], Matthew Figus[3], Noah Goebel[1], Kali Graziano[1], Dana Greene[1], David Nejdl[1], Emmet Nugent[1], Sean Nugent[1], Carmen Soriano Hoyuelos[4], Christopher Repa[1], and Juliet Torres[1]**

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The goal of the experiment is to evaluate the differences between three extant and fossil spiders preserved in amber, using synchrotron x-ray tomography. The propagation phase contrast-based x-ray synchrotron imaging techniques provide detailed 3D images that will allow the group to virtually dissect the fossil species and determine if the changes in the modern samples are due to evolutionary changes in the genus, or alternatively to taphonomical (preservation related) factors. A comparison between the amber-preserved and extant samples will also be conducted to evaluate the effectiveness of amber as a preservation medium. The samples will be scanned using a medium resolution setup to study the general morphology of the specimens. Some areas of anatomical interest will then be scanned at a high resolution (spinnerets and male palps). Each medium resolution scan takes from 40 to 90 minutes depending on the sample horizontal size, and each high resolution scan takes approximately 40 minutes. The use of the synchrotron device at the advanced photon source will show enhanced detail unable to be achieved through conventional methods.

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| **DMS, Inc.**<br>1128 Mary Lane<br>Naperville, IL 60540<br>Tel: 630-983-1704<br>Fax: 630-983-1705<br>darcymicro.wix.com/dmswix | DMS is a manufacturers' representative providing sales and support of the latest technologies in high-resolution microscopic and spectroscopic nanoscience imaging. Products on display include nano-FTIR imaging and spectroscopy systems from Neaspec GmbH. Neaspec is dedicated to delivering innovative solutions for nanoscale optical imaging and spectroscopy for researchers in industry and academic institutions. Neaspec's NeaSNOM – the ultimate nanoanalytic microscopy platform for materials research and photonics – enables optical analysis of complex material systems at visible, infrared, and terahertz frequencies, at a spatial resolution of 10 nm on inorganic and organic samples for both steady-state and time-resolved studies. Also on display will be AFM systems from JPK, the recognized world leader in SPM, with proven highest resolution, lowest noise, and fastest scanning of any SPM systems. JPK Nanowizard 3 systems can be combined with a variety of optical microscopes (upright, inverted, CLSM), and are the only systems providing simultaneous real-time optical and scanning probe imaging of samples. JPK's tip scanning design is ideal for all sample types, including the most challenging soft matter and biological samples and samples in liquids. The new Nanowizard 3 Ultraspeed represents the next generation in SPM, with up to 250 Hz scan rates at highest resolution levels operating in closed loop. |
| **EBARA Technologies Inc.**<br>51 Main Avenue<br>Sacramento, CA 95838<br>Tel: 916-920-5451<br>Fax: 916-925-6654<br>www.ebaratech.com | EBARA is a global innovator/local provider of vacuum pumps and advanced exhaust management solutions for semiconductor, photovoltaic, thin films, and R&D. EBARA's products are backed by a superior global service network. EBARA continues to expand its role by bringing to the market dry vacuum pumps, turbomolecular pumps, and point-of-use abatement. |

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| **FMB Oxford**<br>Unit 1 Ferry Mills<br>Osney Mead<br>Oxford<br>OX2 0ES<br>United Kingdom<br>Tel: +44 1865-320300<br>Fax: +44 1865-320301<br>www.fmb-oxford.com | FMB Oxford is a well-established leader in the supply of beamlines and components to the scientific community, specializing in the design, assembly, test, installation, and commissioning of high-value systems. With more than 20 years' experience in the global synchrotron industry, FMB has built an extensive product range and continues to work with its customers and other experts in the community to expand and develop this range to provide new solutions capable of supporting the increasing demands of the science. FMB Oxford continues to grow and improve every year. The company has the design capability and manufacturing capacity to handle any beamline needs, whether these are for the smallest component or for multiple beamlines. We have a large number of employees with more than 10 years of experience in the field. Most instruments have the benefit of being evolved designs, refined over multiple deliveries. Continual investment in new production, test and design facilities, together with the recruitment and development of key staff, ensures that FMB Oxford remains at the forefront of beamline system supply. Please take a little time to look at our website to view our advanced and expanding range of products. If you can't find what you are looking for, do not hesitate to contact us at enquiries@fmb-oxford.com for further assistance or advice. |
| **Hergo, Inc.**<br>56-01 55th Avenue<br>Maspeth, NY 11378<br>Tel: 888-222-7270<br>Fax: 888-410-0671<br>www.hergo.com | NYC-based Hergo designs, manufactures, and markets an extensive line of functionally engineered modular technical office furniture, including open computer racking systems, lab furniture, data center consoles, 19" enclosures/cabinets and relay racks, flat panel monitor arm solutions, mobile and hospital carts, personal workstations and computer desks, and power and cable management solutions. |
| **Hiwin Corp.**<br>1400 Madeline Lane<br>Elgin, IL 60124<br>Tel: 847-827-2270<br>Fax: 847-827-2291<br>www.hiwin.com | Hiwin Corp. manufactures an extensive line of both linear motion components and complete solutions. With a goal of supplying superior quality at the most competitive prices, the company offers a variety of precision ground and rolled ballscrews, linear guideways, electric actuators, ballscrew-driven stages, and linear motors, with many fully integrated, value-added features. |
| **Huber Diffraction**<br>8344 West Avenue C4<br>Lancaster, CA 93536<br>Tel: 949-391-7755<br>xhuber.com | Huber Diffraction designs and manufactures high-precision positioning systems primarily used for x-ray diffraction in laboratories and at synchrotron beam sources. Other application fields are diffraction experiments with neutrons, laser technology, astronomy, and precise measurement technology. |
| **Instrument Design Technology Ltd.**<br>Unit 2, Turnstone Business Park<br>Mulberry Avenue<br>Widnes<br>WA8 0WN<br>United Kingdom<br>Tel: +44 1516014111<br>Fax: +44 1516014112<br>www.idtnet.co.uk | Instrument Design Technology (IDT) provides the world's synchrotron community with a design-and-build resource in state-of-the-art mechanical high-precision instrumentation. IDT is fully compliant with the latest quality management system, ISO-9001. IDT has core competencies in design and build of complex, state-of-the-art, instruments with motion in multiple degrees of freedom, often working in an ultra-high-vacuum (UHV) environment. Complex optical engineering of x-ray and other beamlines is provided, very often with novel concepts being incorporated. We have a growing reputation for novel monochromators with exceptional proven performance in the field. |
| **KOHZU Precision Co., Ltd.**<br>2-6-15 Kurigi<br>Asao-ku<br>Kawasaki Kanagawa 215-8521<br>Japan<br>Tel: +81 44-981-2131<br>Fax: +81 44-981-2181<br>www.kohzuprecision.com | We provide double-crystal monochromators, vacuum positioning stages, precision rotation stages, alignment stages, and custom products! Please feel free to contact us! |
| **Kurt J. Lesker Company**<br>1925 Route 51<br>Jefferson Hills, PA 15025<br>Tel: 412-387-9007<br>Fax: 412-384-7563<br>www.lesker.com | Manufacturer/distributor of standard and custom high-vacuum and UHV chambers and sub-assemblies, standard and custom vacuum hardware, feedthroughs, and valves, and vacuum pumps of all types. Manufacturer of thin-film deposition systems for sputtering, electron beam, thermal evaporation, and atomic layer deposition. Exclusive North American representative for UHV Design Ltd. motion devices. |

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| **Lake Shore Cryotronics, Inc.**<br>575 McCorkle Boulevard<br>Westerville, OH 43082<br>Tel: 614-891-2244<br>Fax: 614-818-1600<br>www.lakeshore.com | Supporting advanced research since 1968, Lake Shore Cryotronics is a leading innovator in measurement and control solutions for materials characterization under extreme temperature and magnetic field conditions. High-performance product solutions from Lake Shore include Cernox™, germanium, and ruthenium oxide NTC RTDs; silicon and GaAIAs diodes; and platinum PTC RTDs, as well as temperature controllers, temperature monitors, AC resistance bridges, and current sources. Lake Shore serves an international base of scientists at leading university, government, and commercial research institutions and is supported by a global sales network. |
| **Mantis Deposition, Inc.**<br>2839 Wyandot Street<br>Denver, CO 80211<br>Tel: 303-995-5855<br>Fax: 763-390-0047<br>www.mantisdeposition.com | Mantis Deposition and its affiliates are dedicated to the development and manufacture of high-quality systems and components for cutting-edge applications in nanotechnology, thin film deposition, and surface analysis. We enable researchers to both create and analyze the latest materials for advanced materials and device development. Mantis specializes in nanoparticle deposition, RF atom and ion techniques, UHV sputtering, e-beam, MBE, and PLD. We offer a range of nanoparticle deposition sources and systems, sputter cathodes with optional *in situ* tilt, RF atom and RF ion sources, mini e-beam evaporators, organic evaporators, and thermal gas crackers as well as modular UHV and HV R&D deposition systems that can be customized for your application. Our new partner Sigma Surface Science offers state of the art new ESCA and SPM technology with an exciting new hemispherical analyzer, ESCA software, and low-temperature SPM. Mantis and Sigma's highly skilled teams of engineers, physicists, and designers will be happy to work with you on your next materials deposition and analysis challenges. |
| **Materion Electrofusion**<br>44036 South Grimmer Boulevard<br>Fremont, CA 94538<br>Tel: 510-623-1500<br>Fax: 510-623-7600<br>www.materion.com | Leader in the fabrication of CF flange-mounted beryllium windows, water-cooled window assemblies, complex chambers and beam pipes suitable for UHV. Beryllium windows are available in a range of purity levels (98.5% to 99.8%) and thicknesses (6.35 mm to 8 µm). We have extensive in-house fabrication capability and a variety of joining technologies including electron beam welding, vacuum furnace brazing, and cost-effective epoxy bonding. In 2014 we received CERN's CMS Crystal Award for our excellence and innovation in supplying the beam pipe for the CMS Experiment station. |
| **McCrone Group**<br>850 Pasquinelli Drive<br>Westmont, IL 60559<br>Tel: 630-887-7100<br>Fax: 630-887-7764<br>www.mccrone.com | The McCrone Group is internationally recognized as a world leader in microscopy and microanalysis and includes McCrone Associates, McCrone Microscopes & Accessories, and Hooke College of Applied Sciences. McCrone Associates is focused on solving the most difficult materials and particle identification problems. McCrone Associates' scientists consult directly with clients and use the most advanced microscopy techniques and instrumentation to solve their problems. McCrone Microscopes & Accessories is the authorized dealer for Nikon microscopes and measuring equipment, Linkam high-temperature and X-ray stages, the JEOL NeoScope benchtop SEM with EDS, digital imaging systems, and a variety of laboratory supplies. The technical sales representatives at McCrone Microscopes are trusted advisors to scientists worldwide. Hooke College of Applied Sciences provides education and training to scientists worldwide, specializing in SEM and TEM instruction. Hooke College of Applied Sciences has more than 40 specialized short courses in materials analysis. Topics covered include light and electron microscopy, spectroscopy, sample preparation, chemistry and laboratory safety, and image analysis. The online McCrone Atlas of Microscopic Particles is a free, online particle reference for scientists, microscopists, and criminalists engaged in materials characterization and particle identification. |
| **MEWASA AG**<br>Straubstrasse 11<br>CH-7323 Wangs<br>Switzerland<br>Tel: +41 794676518<br>www.mewasa.ch/index_en.htm | For the past 19 years, MEWASA (ISO 9001:2008) has been recognized globally as a highest quality supplier of precision metal welded bellows and bellows assemblies for various HV/UHV beam lines and end station processes at LBNL, NSCL, CERN, SLAC, DESY, PSI, ALBA, NASA, and at many university R&D physics and scientific programs. We are dedicated to process manufacturing excellence. MEWASA's niche is focused on the rapid delivery from prototype to production quantities for our customers' needs. Our scope of business includes in-house design engineering (at no extra charge) to support our customers' design activities, and our proactive involvement in qualifying and validating their bellows specifications, including axial and lateral stress testing for both static and dynamic environments. MEWASA provides a written guarantee/warranty on all our manufactured bellows, and all bellows are leak-tested and certified to 1 x $10^{-9}$ cc (STP)/second (helium). |
| **MICRONIX USA / Symetrie**<br>15375 Barranca Parkway<br>Suite E-106<br>Irvine, CA 92618<br>Tel: 949-480-0538<br>www.micronixusa.com | MICRONIX USA supplies micro- and nano-positioning equipment with a focus on the research market. We offer a full range of precision positioners, hexapods, and control electronics, as well as complete system integration services. Additionally, MICRONIX USA is known as the premier supplier for vacuum to 10-10 torr, clean room, and extreme temperature applications. MICRONIX USA is the exclusive U.S. distributor for Symetrie high-precision hexapods. |

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| **Midwest Vacuum, Inc.**<br>15 Spinning Wheel Road<br>Suite 120<br>Hinsdale, IL 60521<br>Tel: 630-323-5399<br>Fax: 630-323-2142<br>www.midwestvacuum.com | Midwest Vacuum, Inc. has celebrated 30 years of supplying the ultra-high-vacuum community with chambers, valves, pumps, instrumentation, and special solutions to vacuum problems. We also handle helium leak-detection equipment and service. |
| **Molmex Scientific, Inc.**<br>518 Pleasant Street<br>Northampton, MA 01060<br>Tel: 413-587-4000<br>www.molmexscientific.com | Molmex Scientific, Inc. is the North American distributor for the Dectris product line of hybrid pixel detectors and the SAXSLAB Small Angle X-ray Scattering systems for the home lab and synchrotron and represents JJ X-Ray in the United States and Canada for custom beamline solutions. We have more than 30 years of experience in the fabrication of x-ray scattering instrumentation and are located in Northampton, MA. |
| **Navitar**<br>200 Commerce Drive<br>Rochester, NY 14623<br>Tel: 585-359-4000<br>www.navitar.com | Navitar designs, develops, manufactures, and markets precision optical solutions for a wide variety of industries, including biomedical and life sciences, defense and security, industrial imaging, and projection, spanning from standard video to 4K planetarium lenses. Offerings include high-magnification zoom and fixed lenses, such as the new MicroMate 3:1 Zoom for 4/3" sensors, MagniStar double-sided telecentric lenses, microscope objectives, modular microscopy components, and custom lens design. Applications for Navitar optics include machine vision, automation, assembly, imaging, testing, measuring, metrology, biometrics, telecommunications, photonics R&D, noncontact measurement, simulation, planetarium, amusement, and more. |
| **Newark**<br>1400 Charles Avenue<br>Algonquin, IL 60102<br>Tel: 847-757-0118 | www.newark.comNewark element14 is a high-service distributor of technology products, services, and solutions for electronic system design, maintenance, and repair. Newark brings together the latest products, services, and development software, all connected to an innovative online engineering community where purchasers and engineers can access peers and experts, a wide range of independent technical information, and helpful tools. Whether researching a new technology, designing an electronic product, or looking for parts to repair an existing system, we are the trusted global source to find the answers and parts you need to keep your project on the fast track, right from the start. Additional information is available at www.newark.com Keysight Technologies (www.keysight.com) is a global electronic measurement technology and market leader helping to transform its customers' measurement experience through innovations in wireless, modular, and software solutions. Keysight's electronic measurement instruments, systems, software and services are used in the design, development, manufacture, installation, deployment and operation of electronic equipment. |
| **nPoint, Inc.**<br>3030 Laura Lane<br>Suite 100<br>Middleton, WI 53562<br>Tel: 608-824-1770<br>Fax: 608-824-1774<br>www.npoint.com | Designs and manufactures nano-positioning tools for OEM and research applications. Products include piezo-actuated flexure stages and digital control electronics. Nano-positioning systems provide closed-loop translation in the X, XY, XYZ, and tip/tilt. Enables high-speed, high-resolution, sub-nanometer precision for the optics, semiconductor, defense, aerospace, and biological industries. |
| **Oerlikon Leybold Vacuum**<br>5700 Mellon Road<br>Export, PA 15632<br>Tel: 724-327-5700<br>www.oerlikon.com/leyboldvacuum/ | Oerlikon Leybold Vacuum is one of the world's leading suppliers of vacuum equipment, components, and systems to the semiconductor, industrial coating, metallurgy, solar, R&D, analytical, and scientific instrument markets. Our wide range of products include conventional ceramic bearing turbomolecular pumps, magnetic bearing turbomolecular pumps, oil-sealed rotary vane mechanical pumps, dry compressing scroll pumps, vacuum gauges, helium leak detectors, cryopumps, mechanical boosters and diffusion pumps. We also offer extensive engineering services for customized pumping systems, including our line of UNIVEX high vacuum deposition systems for PVD applications, such as RF/DC sputtering, thermal resistance evaporation, electron beam evaporation and ion beam assisted deposition. In addition, we also provide repair services for all brands of vacuum pumps. Our Vacuum Academy offers training courses in fundamental vacuum principles, practical leak detection, instrumentation, or maintenance and repair of your specific vacuum pumps. We can also customize a vacuum course to meet your specific needs. The very affordable courses are offered either at our facility in Export, Pennsylvania, or on-site at your facility. For more information or to receive a quote on any of our products or services, please contact us at info.vacuum.ex@oerlikon.com. |
| **Olympus America Inc.**<br>3500 Corporate Parkway<br>Center Valley, PA 18034<br>Tel: 484-896-5844 | Olympus Scientific Solutions Americas, a part of the global Olympus life science and industrial instrumentation business, delivers solutions for clinical, educational, and research microscopes, nondestructive testing equipment, and analytical instruments. Designed to contribute to the safety, security, quality, and productivity of society, Olympus strives to enhance people's lives every day. |

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| **OMS Motion, Pro-Dex**<br>15201 NW Greenbrier Parkway, B-1<br>Beaverton, OR 97006<br>Tel: 800-707-8111<br>Fax: 503-629-0688<br>www.OMSmotion.com | OMS Motion is committed to its customers' success, providing world-class motion control solutions in applications of all types. Whether it's vital medical analysis, precise semiconductor equipment or advanced scientific research, engineers depend on the proven reliability that OMS provides. OMS Motion, located in Beaverton, Oregon, is a division of Pro-Dex, Inc., a publicly traded company, and has been successfully producing motion controllers for more than 30 years. OMS was founded as Oregon Micro Systems (OMS) and evolved from patented motion control technology that provides superior advantages in the industry. OMS built a strong reputation in the market for reliable and quality motion control products backed by superior customer service and technical support. OMS motion controllers control up to 10 axes of motion on a single controller. Applications of these controllers include semiconductor and medical equipment, life sciences, and factory automation markets, as well as major research projects at facilities around the world. In addition to creating multi-axis motion controllers, OMS offers motors, drives and accessories that are often tailor-made to support specific applications. OMS provides the experience, proven reliability and accuracy that gain the trust and confidence of design engineers worldwide. |
| **PI (Physik Instrumente) L.P.**<br>16 Albert Street<br>Auburn, MA 01501<br>Tel: 508-832-3456<br>Fax: 508-832-0506<br>www.pi-usa.us | PI/PI miCos is a leading supplier of high-precision positioning and motion-control products for beam-line instrumentation, nanotechnology and microscopy applications. PI employs more than 750 people worldwide and provides the broadest and deepest portfolio of motion technologies, from piezo ceramics to air-bearing stages. Two of our products were selected for the Mars Rover Curiosity science lab because of their excellent performance and reliability. |
| **Princeton Instruments**<br>3660 Quakerbridge Road<br>Trenton, NJ 08619<br>Tel: 609-587-9797<br>Fax: 609-587-1970<br>www.princetoninstruments.com | Princeton Instruments designs and manufactures high performance CCD, ICCD, and EMCCD cameras, spectrographs, and optics-based solutions for the scientific research, industrial imaging, and OEM communities. We take pride in partnering with our customers to solve their most challenging problems in unique, innovative ways. |
| **Rayonix, L.L.C**<br>1880 Oak Avenue<br>Suite 120<br>Evanston, IL 60201<br>Tel: 847-869-1548<br>Fax: 847-869-1587www.rayonix.com/ | Rayonix, L.L.C. is the leading developer of innovative x-ray detector technologies. Five Rayonix high-speed x-ray detector systems are now installed at APS! The Rayonix frame-transfer CCD facilitates data collection at up to 140 frames/second. These high-speed x-ray detectors have no count rate limitation. New detector designs, including the MX170-HS with a center hole, allow the direct beam to pass through, for simultaneous SAXS/WAXS data collection.Talk to us for custom versions of our detectors for specific needs. |
| **Renishaw, Inc.**<br>5277 Trillium Boulevard<br>Hoffman Estates, IL 60192<br>Tel: 847-286-9953<br>Fax: 847-286-9974<br>www.renishaw.com | Renishaw, Inc. is a global company with core skills in measurement, motion control, spectroscopy, and precision machining. The company develops innovative products that significantly advance customers' operational performance—from improving manufacturing efficiencies and raising product quality to maximizing research capabilities and improving the efficacy of medical procedures. |
| **Rigaku Innovative Technologies, Inc.**<br>1900 Taylor Road<br>Auburn Hills, MI 48326<br>Tel: 248-232-6400<br>Fax: 248-232-6500<br>www.Rigaku.com | With more than 25 years of producing multilayer x-ray optics, Rigaku Innovative Technologies has introduced many new developments in instruments and optics to the synchrotron community. Our division of Rigaku designs and builds multilayer optics and rotary vacuum feedthroughs for synchrotron beamlines and end stations. Come learn about our new and innovative multilayer material systems and advanced focusing monochromators for beamlines. Also new is our 2-D Kratky SAXS camera that can be mounted on a beamline end station. Our capabilities include multiple-stripe double monochromators, graded d-spacing structures, and coatings on complex optical shapes. We have recently expanded our coatings capabilities and commissioned our new large format in-line deposition system capable of multilayer structures on 700 mm diameter substrates. Based in Michigan with an extensive portfolio of x-ray optic patents, our renowned team of scientists at Rigaku-Osmic can help solve your most difficult multilayer optic applications. |
| **SAES Getters USA Inc.**<br>1122 E. Cheyenne Mountain Boulevard<br>Colorado Springs, CO 80906<br>Tel: 719-576-3200<br>Fax: 719-576-5025<br>www.saesgetters.com | SAES Group has offered a wide range of compact UHV pumping solutions based on non-evaporable getter (NEG) technology since the 1970s. In the 1990s, SAES pioneered pumps based on sintered porous disks, improving the pumping performance while achieving superior mechanical stability, thus drastically reducing the chance of particle release. Currently, SAES offers a variety of compact, mechanically stable, and simple-to-operate pumps with unparalleled pumping performance in HV, UHV, and XHV. |

**Scientific Instruments, Inc.**
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Fax: 561-881-8556
www.scientificinstruments.com

Scientific Instruments, Inc., is a manufacturer of cryogenic sensor and instrumentation products and is also involved in the aerospace, liquid natural gas, industrial, and aircraft industries.

**SGX Sensortech (MA) Ltd.**
Sirius House, Watery Lane
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High Wycombe
HP10 0AP
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Fax: +44 1628-533034
www.sgxsensortech.com

SGX Sensortech has a distinguished heritage in the manufacture of silicon drift detectors (SDD) and Si(Li) detectors. Previously known as e2v scientific and Gresham Scientific, SGX specializes in producing detectors from standard designs through customized assemblies to complex multi-element detectors. All detectors are designed to meet the highest specifications, including the largest single active area (170 mm2) available of its kind.

- Multi-element silicon drift detectors: SDD SGX Sensortech offers custom multi-sensor silicon drift detector design capability for special applications. Detectors are available with discrete or monolithic arrays of sensors covering a range of active areas and window types, all built for optimum performance.
- Customized multi-element detectors: SDD designs are available to suit geometrical constraints and special applications. Applications include x-ray fluorescence (XRF), micro x-ray fluorescence (μXRF), total reflection x-ray fluorescence (TXRF), particle induced x-ray emission (PIXE), extended x-ray absorption fine structure (EXAFS), and x-ray absorption near edge structure (XANES).

**SPECS Surface Nano Analysis, Inc.**
20 Cabot Boulevard
Suite 300
Mansfield, MA 02048
Tel: 508-618-1292
Fax: 508-618-1334
www.specs.com

SPECS manufactures cutting-edge systems and components for surface analysis in UHV, based on methods such as XPS, UPS, AES, ISS, STM, LEEM/PEEM, LEED, SIMS, SNMS, and HREELS. We offer a variety of sources for deposition, excitation, and charge neutralization, as well as analyzers, monochromators, and research microscopes like LEEM and STM. A strong focus of our work is on customized systems combining thin-film preparation (MBE) with spectroscopic and microscopic options.

**Spectral Instruments**
420 N. Bonita Avenue
Tucson, AZ 85745
Tel: 520-884-8821
www.specinst.com

Spectral Instruments specializes in the development and manufacture of cooled CCD cameras for demanding low-light applications. We provide the finest low noise (<3e-), high sensitivity, -110°C cooling, 16-bit CCD cameras that current technology can offer. Optical inputs can include windows with custom coatings through fiber optic tapers or faceplates. Readout options are designed to be flexible and customized to the application. Custom CCD designs and OEM camera production are available.

**Starrett Tru-Stone Tech**
1101 Prosper Drive
Waite Park, MN 56387
Tel: 320-251-7171

Precision granite design collaboration, production, and assembly services. Starrett Tru-Stone Technologies specializes in precision granite machine bases and assemblies. We partner with you to add expertise in design collaboration; high-quality, on-time manufacturing; and complex assembly for your precision base. In addition to granite bases, Tru-Stone technicians install precision rails, air mounts, brackets, pneumatic systems, and cabling. Custom stands are also provided. Materials beyond granite include ceramic and carbon fiber.

**TDK-Lambda Americas High Power Division**
405 Essex Road
Neptune, NJ 07753
Tel: 732-922-9300
Fax: 732-922-1441
www.us.tdk-lambda.com/hp/

TDK-Lambda Americas High Power Division, located in Neptune, New Jersey, is a manufacturer of programmable, high-density power supplies. The Genesys ™ series of programmable power supplies has the highest density in power levels from 750W through 15KW, with output ranges up to 600V and 1,000A. TDK-Lambda Genesys™ Series of programmable power supplies has the highest density, making the Genesys™ Series the most complete set of platforms with identical user interfaces. Platforms include 750 W, 1500 W, 3.3/5 kW and 10/15 kW output. Outputs available range up to 600 V and 1,000 A. In addition to saving space, Genesys™ power supplies contain extensive standard features that provide a new level of performance and flexibility. Also now available, an optional LXI Certified LAN interface provides flexible system integration and functionality.

**Tek-Matic Inc.**
7324 Forest Hills Road
Loves Park, IL 61111
Tel: 815-282-1775
Fax: 815-282-1862
www.tek-matic.com

Tek-Matic partners with lab automation companies providing precision motion, sensing, and vision camera products, assemblies and systems. Tek-Matic provides sizing, selection, logistics, training, and unparalleled support. Available products include USB/E-net based stepper/servo products, measurement sensors, positioning systems and actuators, piezo motors/stages for sample and optics manipulation, precision microscope stages, and OEM vision cameras.

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| **The Fredericks Company/TELEVAC**<br>2400 Philmont Avenue<br>Huntingdon Valley, PA 19006<br>Tel: 215-947-2500<br>Fax: 215-947-7464<br>www.TELEVAC.com | The Fredericks Company/TELEVAC has been providing innovative vacuum solutions for more than 80 years. Our instruments and sensors are regarded as the gold standard in the vacuum industry. TELEVAC's double inverted magnetron patented technologies have revolutionized the vacuum industry, providing rugged and reliable performance cold cathodes and instruments. We have been developing new products to serve the vacuum industry in many of its diverse applications, including national labs, heat treating, thin-film coating, and many others. For more information, please contact us at 215-947-2500 or visit our website at www.TELEVAC.com. |
| **Toyama Co., Ltd**<br>4-13-16, Hibarigaoka<br>Hibarigaoka<br>Zama-Shi<br>Kanagawa 252-0003<br>Japan<br>Tel: +44 7802832377<br>www.toyama-en.com | Toyama can provide a full range of design, manufacturing, installation, and commissioning services for synchrotron beamline and accelerator projects. |
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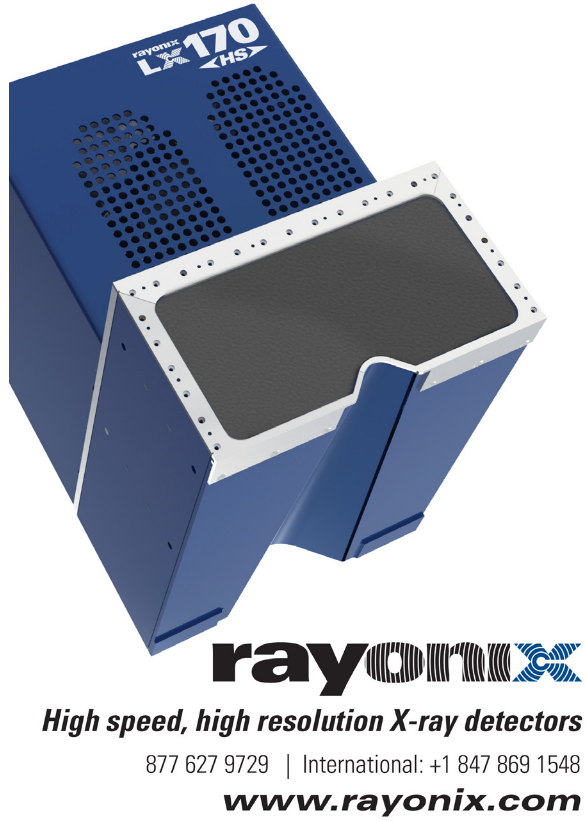
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# 2015
## APS/CNM Users Meeting

# GENERAL INFORMATION

# Practical Matters

## Locations

General sessions will be held in the Auditorium on the first floor of the APS conference center, Bldg. 402. Vendor exhibits will be in the center's lower level and the atrium on the main level. Workshop locations are listed on the Comprehensive Program and posted at the registration desk.

## Meals

The conference fee includes a continental breakfast and coffee breaks each day and the poster session reception. Buffet lunches will be served Monday through Wednesday in the tent outside of the Gallery on the lower level. If you pre-ordered lunches on your registration form, you will receive your tickets when you receive your meeting materials. (A limited number of extra lunch tickets are available for purchase at the registration desk.) Banquet tickets are nonrefundable. The Argonne cafeteria will be open for lunch; the Guest House restaurant it is open for dinner. The 401 Grill will be open for dinner throughout the meeting. A list of nearby restaurants is available at www.aps.anl.gov/About/Visiting/Restaurants.

## Telephones and Messages

Messages for you can be left at the registration desk; the telephone numbers there are 630.252.9580 and 630.252.9581. The messages will be posted on a bulletin board by the entrance to the Auditorium. If you need to make a telephone call, a pay phone is located downstairs near the restrooms at the back of the Gallery, lower level. If you need to send or receive a fax, a fax machine is located in the APS User Office (Bldg. 401, Rm. B1154). The number of this machine is 630.252.9250.

## Transportation

Conference staff can make limousine reservations for you during the meeting as long as you make your request before 1:00 pm on Wednesday.

## ATM

An automated teller machine is located in Bldg. 233, behind the Argonne cafeteria. This machine accepts the following cards: American Express, Discover/Novus, The Exchange, Master Card, Plus, Visa, and 24 Access.

# Computer Access

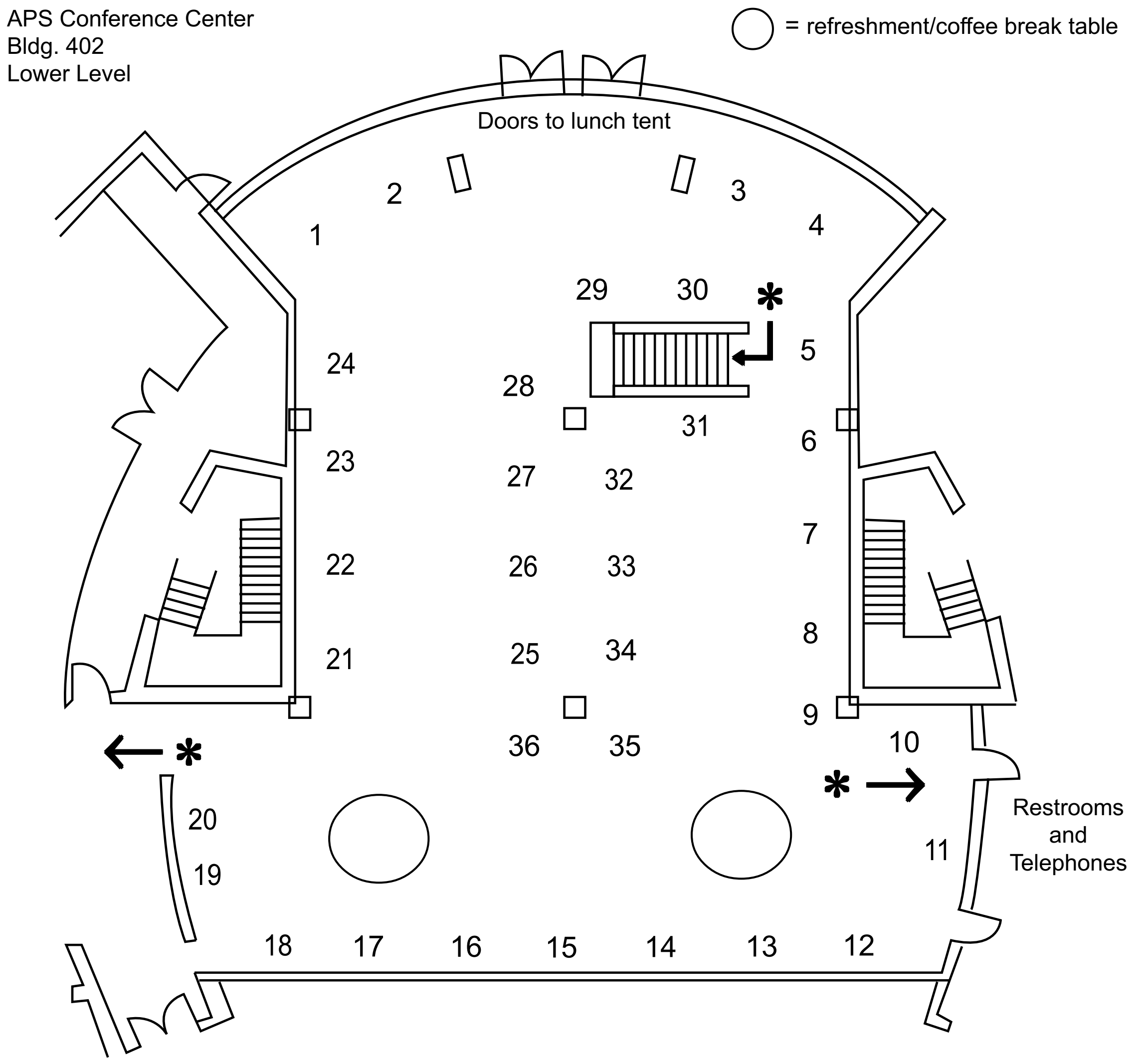
Public computer terminals are available in the Bldg. 401 atrium behind the silver wall. Wireless access is also available in the Conference Center.

To use your laptop computer on the APS wireless networks, complete the following steps:

1. Open the wireless connection on your computer (either the 401 or 402 networks).
2. Read and accept the Argonne internet access policy, which will appear as a Web page on your desktop. After you click "accept," a registration Web page will appear.
3. The registration web page asks you for the following information:
    a. First and last name
    b. E-mail address
    c. Building and room where you will be located (use 402 conference center)
    d. Phone number where you can be reached on site (use 630-252-9090)
    e. Name of person you are visiting or conference you are attending
    f. Home institution
    g. Do you need to send e-mail directly to an off-site server?
    h. What is the duration of this registration? (You'll be given choices.)

If you have problems, please contact someone in the APS User Office (B1154, located immediately off the Conference Center atrium).

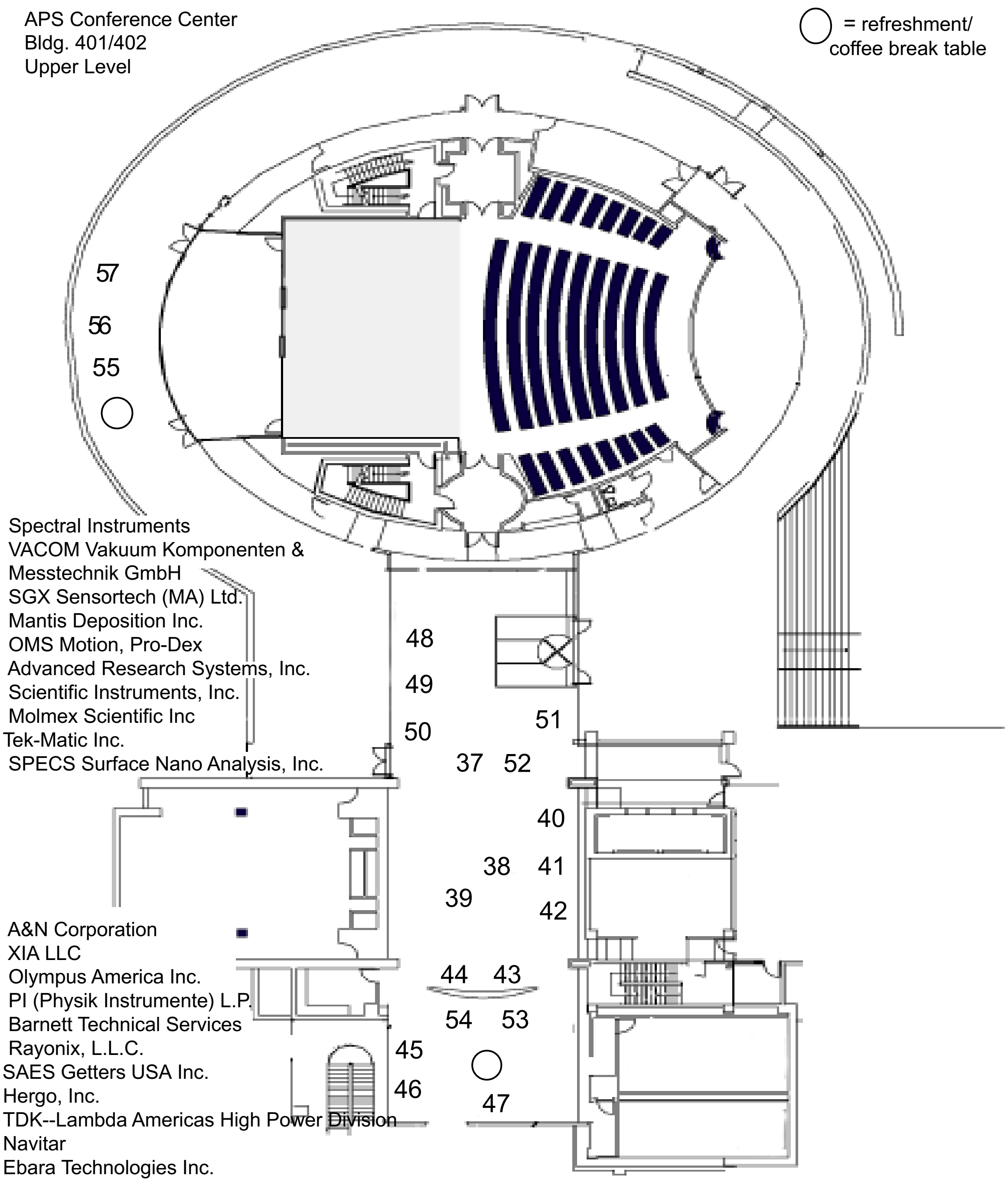
# Exhibitor Locations – 2015 APS/CNM Users Meeting
# Argonne National Laboratory




| | | |
|---|---|---|
| 1. attocube systems Inc. | 13. Rigaku Innovative Technologies | 25. COSMOTEC, Inc. |
| 2. Midwest Vacuum, Inc. | 14. Newport Corporation | 26. DMS, Inc. |
| 3. Instrument Design Technology | 15. KOHZU Precision Co. Ltd. | 27. *withdrawn* |
| 4. MICRONIX USA / Symetrie | 16. Vacuum One | 28. MEWASA AG |
| 5. Huber Diffraction | 17. BellowsTech, LLC | 29. Advanced Design Consulting USA, Inc. |
| 6. Applied Diamond Inc. | 18. Newark | 30. Carl Zeiss Laser Optics GmbH |
| 7. CINEL Strumenti Scientifici SRL | 19. McCrone Group | 31. Bruker |
| 8. Princeton Instruments | 20. Kurt J. Lesker Company | 32. Lake Shore Cryotronics |
| 9. Aerotech, Inc. | 21. Oerlikon Leybold Vacuum | 33. Renishaw Inc. |
| 10. The Fredericks Co. / TELEVAC | 22. Toyama Co., Ltd. | 34. Materion Electrofusion |
| 11. Starrett Tru-Stone Tech | 23. nPoint, Inc. | 35. Canberra |
| 12. Hiwin Corp. | 24. FMB Oxford | 36. Alan Burrill Technical Sales |

* UPSTAIRS/ATRIUM EXHIBITORS PLUS REFRESHMENT TABLES

# Exhibitor Locations – 2015 APS/CNM Users Meeting
# Argonne National Laboratory




37. Spectral Instruments
38. VACOM Vakuum Komponenten & Messtechnik GmbH
39. SGX Sensortech (MA) Ltd.
40. Mantis Deposition Inc.
41. OMS Motion, Pro-Dex
42. Advanced Research Systems, Inc.
43. Scientific Instruments, Inc.
44. Molmex Scientific Inc
45. Tek-Matic Inc.
46. SPECS Surface Nano Analysis, Inc.

47. A&N Corporation
48. XIA LLC
49. Olympus America Inc.
50. PI (Physik Instrumente) L.P.
51. Barnett Technical Services
52. Rayonix, L.L.C.
53 SAES Getters USA Inc.
54 Hergo, Inc.
55. TDK--Lambda Americas High Power Division
56 Navitar
57 Ebara Technologies Inc.

Additional exhibitors on the Gallery level.

# 2015 APS/CNM Users Meeting - Schedule at a Glance

## Monday, May 11th

**Vendor exhibits open daily Monday through Wednesday in the Gallery (lower level) and the Atrium (first floor). Break time snacks offered in all locations!**

**Opening Session** (*Bldg. 402, APS Auditorium*)

| | |
|---|---|
| **7:30** | **Registration Open** |
| **7:30** | **Welcome Coffee** |
| 8:50 - 9:00 | Eric Landahl, APSUO Chair, Welcome |
| 9:00 - 9:20 | Peter Littlewood, ANL Director, Welcome from the Laboratory |
| 9:20 - 9:40 | Harriet Kung, DOE Office of Basic Energy Sciences, The DOE Perspective |
| 9:40 - 9:45 | Seungbum Hong, CNM UEC Chair, Introduction of Keynote |
| 9:45 - 10:25 | **Keynote Speaker**: John Rogers, University of Illinois Urbana-Champaign |
| **10:25 - 11:00** | **Coffee Break** |
| 11:00 - 11:25 | Stephen Streiffer, Director, APS |
| 11:25 - 11:45 | Andreas Roelofs, Interim Director, CNM |
| 11:45 - 12:00 | Susan Strasser, NUFO/ARIA |
| **12:00 - 1:30** | **Lunch (Gallery lower level)** |

**Parallel Plenary Sessions**

| APS (*Bldg. 402, APS Auditorium*) | | CNM (*Bldg. 402, Rooms E1100-1200*) | |
|---|---|---|---|
| 1:15- 1:55 | Keynote: Wenlu Zhu | 1:30 - 2:15 | Keynote: Vlad Shalaev |
| 1:55 - 2:35 | Compton Award Presentation/Talk | 2:15 - 2:45 | Teri Odom |
| 2:35 - 2:55 | Andrew Ulvestad, Invited Student | 2:45 - 3:15 | Heinrich Jaeger |
| **2:55- 3:25** | **Coffee Break** | **3:15 - 3:35** | **Coffee Break** |
| 3:25 - 3:55 | Robert Leheny | 3:35 - 3:45 | CNM UEC Update |
| 3:55 - 4:25 | Reeju Pokharel | 3:45 - 4:15 | Alex Martinson |
| 4:25 - 4:55 | David Vine | 4:15 - 4:45 | Jonathan J. Foley, IV |
| | | 4:45 - 5:00 | Vuk Brajuskovic, Invited Student |

**Coffee breaks are offered in 3 areas:**
the Gallery on the lower level, the Atrium,
and outside E1100/1200.
**PLEASE BE SURE TO VISIT THE EXHIBITORS IN EACH AREA!**

**Poster Session set up will take place on Tuesday, May 12. For poster set up, shuttle buses and vans will run between the APS, the Guest House, and the TCS** ***between 12 noon and 2:00 pm*** **.**

| | |
|---|---|
| **5:00 - 9:00** | **Social Event: Banquet at the Garfield Park Conservatory, (buses depart promptly at 5:15 pm from the front of APS and the AGH)** |

## Tuesday, May 12th

| | |
|---|---|
| **7:30** | **Welcome Coffee** |
| **8:00** | **Registration Open** |
| **12:00 - 1:30** | **CNM Users Executive Committee Meeting** (***Bldg. 401, Room B5100***) |
| **12:00 - 1:30** | **APS Partner User Council Meeting** (***Bldg. 401, A5000 Gallery***) |
| **5:00** | **DCS Tour at Sector 35** |
| **5:30 - 8:00** | **Poster Session and Reception (Bldg. 240 TCS--bus transportation provided between APS, Guest House, and TCS)** |

**CNM WK 1: Nanophotonic Structures, Surfaces, and Composites for Radiative Control (Bldg. 401, Room A5000)**

| | |
|---|---|
| 8:30 - 8:45 | Welcome & Introductory Remarks |
| 8:45 - 9:25 | Marco Loncar |
| 9:25 - 10:05 | George Schatz |
| **10:05 - 10:35** | **Coffee Break** |
| 10:35 - 11:15 | Yugang Sun |
| 11:15 - 12:00 | Zhiqun Lin |
| **12:00 - 1:30** | **Lunch (Gallery lower level)** |
| 1:30 - 2:10 | Peng Zhang |
| 2:10 - 2:50 | Shaung Fang Lim |
| **2:50 - 3:20** | **Coffee Break** |
| 3:20 - 4:00 | Stephen Gray |
| 4:00 - 4:40 | Nathaniel Stern |
| 4:40 - 5:00 | Wrap-up and Concluding Remarks |

**APS WK2: High-energy Resolution Inelastic X-ray Scattering Workshop (*Bldg. 402, APS Auditorium*)**

| | |
|---|---|
| 8:30 - 8:35 | Welcome & Introductory Remarks |
| 8:35 - 8:45 | Ayman Said |
| 8:45 - 9:25 | Frank Weber |
| 9:25 - 10:05 | Mathieu Le Tacon |
| **10:05 - 10:35** | **Coffee Break** |
| 10:35 - 11:15 | Jiawang Hong |
| 11:15 - 11:55 | Charles W. Myles |
| **12:00 - 1:30** | **Lunch (Gallery lower level)** |
| 1:30 - 2:10 | John Budai |
| 2:10 - 2:50 | Jason Hancock |
| **2:50 - 3:20** | **Coffee Break** |
| 3:20-4:00 | Jung-Fu Lin |
| 4:00 - 4:40 | Nicholas Butch |
| 4:40 - 5:00 | Wrap-up and Concluding Remarks |

**APS WK3: Developing Synchrotron Sample Environments to Study Next-generation Field-driven Device Physics (*Bldg. 401, Room A1100*)**

| | |
|---|---|
| 8:30 - 9:00 | Philip Ryan |
| 9:00 - 9:40 | Markys Cain |
| 9:40 - 10:00 | Glenn Martyna |
| **10:00 - 10:35** | **Coffee Break** |
| 10:35 - 11:15 | Paul Evans |
| 11:15 - 11:55 | Jacob Jones |
| **12:00 - 1:30** | **Lunch (Gallery lower level)** |
| 1:30 - 2:10 | Siddarth Saxena |
| 2:10 - 2:50 | Peter Fisher |
| **2:50 - 3:20** | **Coffee Break** |
| 3:20 - 4:00 | Xavi Marti |
| 4:00 - 4:40 | SaeHwan Chun |

**CNM WK4: Exploring the Flatland of 2D Materials for Tribological Manipulation (*Bldg. 401, Room E1100/1200*)**

| | |
|---|---|
| 8:30 - 8:45 | Welcome & Introductory Remarks |
| 8:45 - 9:25 | Robert Carpick |
| 9:25 - 10:05 | James Batteas |
| **10:05 - 10:35** | **Coffee Break** |
| 10:35 - 11:15 | Elena Polyakova |
| 11:15-12:00 | Alex Smolyanitsky |
| **12:00 - 1:30** | **Lunch (Gallery lower level)** |
| 1:30 - 2:10 | Yury Gogotsi |
| 2:10 - 2:50 | Christopher Muratore |
| **2:50 - 3:20** | **Coffee Break** |
| 3:20 - 4:00 | Nikhil Koratkar |
| 4:00 - 4:40 | Diana Berman |
| 4:40 - 5:00 | Wrap-up and Concluding Remarks |

## Wednesday, May 13th

| | |
|---|---|
| **7:30** | **Welcome Coffee** |
| **8:00** | **Registration Open** |
| **12:00 - 1:30** | **APSUO Steering Committee Meeting** (***Bldg. 401, Fifth Floor Gallery***) |

**APS WK 6: APS Upgrade: The First Experiments MORNING (*Bldg. 402, APS Auditorium*)**

| | |
|---|---|
| 8:30 - 12:00 | Open Forum Workshop |
| **10:05 - 10:35** | **Coffee Break** |
| **12:00 - 1:30** | **Lunch (Gallery lower level)** |

**APS WK8: *In situ* X-ray Characterization of Microstructure during Manufacture MORNING (*Bldg. 401, Room A5000*)**

| | |
|---|---|
| 8:30 - 8:40 | Welcome & Introductory Remarks |
| 8:40 - 9:20 | Amy Clarke |
| 9:20 - 10:00 | Jon Emery |
| **10:00 - 10:30** | **Coffee Break** |
| 10:30 - 11:10 | Ruipeng Li |
| 11:10 - 11:50 | Aaron Stebner |
| 11:50 - 12:00 | Wrap-up and Concluding Remarks |
| **12:00 - 1:30** | **Lunch (Gallery lower level)** |

**APS WK9: Workshop on 119Sn Nuclear Resonant Scattering at the APS MORNING (*Bldg. 401, Room E1100/1200*)**

| | |
|---|---|
| 8:30 - 8:35 | Welcome & Introductory Remarks |
| 8:35 - 9:05 | Ercan Alp |
| 9:05 - 9:45 | John Tse |
| 9:45 -10:25 | Raphael Hermann |
| **10:25 - 10:35** | **Coffee Break** |
| 10:35 - 11:15 | Mathieu Roskosz |
| 11:15 - 11:55 | W. Robert Scheidt |
| 11:55 - 12:00 | Wrap-up and Concluding Remarks |
| **12:00 - 1:30** | **Lunch (Gallery lower level)** |

**APS WK3 cont'd: Developing Synchrotron Sample Environments to Study Next-generation Field-driven Device Physics MORNING (Bldg. 432, C010 Conf. Room)**

| | |
|---|---|
| 9:00 - 9:40 | Martin Holt |
| 9:40 - 10:20 | Jian Liu |
| 10:20 - 11:00 | Raegan Johnson |
| 11:00-11:40 | Sean McMitchell |
| 11:40 - 12:20 | Yong Choi |
| 12:20 | Wrap-up |
| **12:00 - 1:30** | **Lunch (Gallery lower level)** |

**APS/CNM WK7: Experimental and Computational Challenges of *in situ* Multimodal Imaging of Energy Materials (*Bldg. 401, Room A1100*)**

| | |
|---|---|
| 8:30 - 8:40 | Welcome & Introductory Remarks |
| 8:40 - 9:30 | Nigel Browning |
| 9:30 - 10:00 | Haimei Zheng |
| **10:00 - 10:35** | **Coffee Break** |
| 10:35 - 11:05 | Chongmin Wang |
| 11:05 - 11:35 | Scott Warren |
| 11:35 - 12:00 | Jeffrey Greeley |
| **12:00 - 1:30** | **Lunch (Gallery lower level)** |
| 1:30 - 2:20 | Paul Fenter |
| 2:20 - 2:50 | Jiajun Wang |
| **2:50 - 3:20** | **Coffee Break** |
| 3:20-3:50 | Raymond Osborn |
| 3:50 - 4:20 | Todd Turner |
| 4:20 - 4:50 | Mark Hereld |
| 4:50 - 5:00 | Wrap-up and Concluding Remarks |

**APS WK10: Application of Synchrotron X-ray Scattering Techniques to Nuclear Materials AFTERNOON (*Bldg. 401, Room A5000*)**

| | |
|---|---|
| 1:30 - 1:40 | Welcome & Introductory Remarks |
| 1:40 - 2:10 | Jim Stubbins |
| 2:10 - 2:40 | Meimei Li |
| 2:40- 3:10 | Mohamed Elbakhshwan |
| **3:10 - 3:30** | **Coffee Break** |
| 3:30 - 4:50 | APS Radiation Safety Officer |
| 4:50 - 5:00 | Round Table Discussion |

**APS WK11: Tracking Electronic and Structural Dynamics in Proteins and Materials at Sector 14 AFTERNOON (*Bldg. 401, Room E1100/1200*)**

| | |
|---|---|
| 1:30 - 1:40 | Welcome & Introductory Remarks |
| 1:40 - 2:10 | Hyun Sun Cho |
| 2:10 - 2:40 | Xiaoshan Xu |
| 2:40- 3:10 | Marius Schmidt |
| **3:10 - 3:30** | **Coffee Break** |
| 3:30 - 4:00 | Stephen Durbin |
| 4:00 - 4:30 | Sebastian Westenhoff |
| 4:30 - 5:00 | Aaron Lindenberg |
| 5:00 | Wrap-up and Concluding Remarks |

## Thursday, May 14th

**(pre-registration required)**

**CNM Short Courses**

| | |
|---|---|
| **Short Course A** | Bldg. 212, A157 (all day) |
| **Short Course B** | Bldg. 440, main lobby (morning) |
| **Short Course C** | Bldg. 440, main lobby (morning) |
| **Short Course D** | Bldg. 440, main lobby (afternoon |

# 2015 APS/CNM USERS MEETING

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